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A PRACTICAL TREATISE  
ON THE  
MANUFACTURE OF SOAP AND CANDLES:

BASED UPON THE  
MOST RECENT EXPERIENCES IN THE SCIENCE  
AND THE PRACTICE;  
COMPRISING THE  
CHEMISTRY, THE RAW MATERIALS, THE MACHINERY AND UTENSILS,  
AND VARIOUS PROCESSES OF MANUFACTURE, INCLUDING  
A GREAT VARIETY OF FORMULAS.

EDITED CHIEFLY FROM THE GERMAN OF  
DR. C. DEITE, A. ENGELHARDT, DR. C. SCHAEGLER, AND OTHERS.  
WITH ADDITIONS AND  
LISTS OF AMERICAN PATENTS RELATING TO THESE SUBJECTS.

BY  
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EDITOR OF "A PRACTICAL TREATISE ON ANIMAL AND VEGETABLE FATS AND OILS;"  
ONE OF THE EDITORS OF "THE TECHNO-CHEMICAL RECIPE BOOK."

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GEORGE J. WHITHED  
WOBURN, MASS.

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## PREFACE.

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THE magnitude of the soap industry, the importance of the trade, and the enormous capital embarked in it, as well as the wonderful relation which it bears with regard to the most important links in the chain of chemical industry, are not often sufficiently estimated. The distinguished chemist, Justus von Liebig, in his "Familiar Letters on Chemistry," says:—

"The quantity of soap consumed by a nation would be no inaccurate measure whereby to estimate its wealth and civilization. Political economists, indeed, will not give it this rank; but whether we regard it as joke or earnest, it is not the less true that, of two countries with an equal amount of population, we may declare with positive certainty that the wealthiest and most highly civilized is that which consumes the greatest weight of soap. This consumption does not subserve sensual gratification, nor depend upon fashion, but upon the feeling of the beauty, comfort, and welfare attendant upon cleanliness; and a regard to this feeling is coincident with wealth and civilization. The rich in the middle ages, who concealed a want of cleanliness in their clothes and persons under a profusion of costly scents and essences, were more luxurious than we are in eating and drinking, in apparel and horses; but how great is the difference between their days and our own, when a want of cleanliness is equivalent to insupportable misery and misfortune?"

It is, however, only in the most modern times that the soap manufacture has attained the extraordinary magnitude which now



distinguishes this branch of trade. Various circumstances have contributed to produce it, the principal one, however, being the development of the manufacture of soda, which freed the soap industry from its dependence on the uncertain and limited supply of barilla and kelp, and caused it to make such strides as could not have been anticipated by the most sanguine. The manufacture of soap has, on the other hand, been a powerful stimulus to the preparation of soda and of the important secondary product, chloride of lime (bleaching powder), which are so intimately allied with almost all branches of the chemical art. Thus soap occupies one of the most important pages in the history of applied chemistry. The increase in the consumption of this article has led, moreover, to the discovery of new materials for its production; it has opened new channels to commerce, and thus it has become the *means* as well as the *mark* of civilization. Almost simultaneously with the employment of soda, the oils of the cocoanut and the palm have been introduced into the manufacture of soap, and there can be no doubt that the development of the trade in palm-oil has largely contributed to the abolition of the iniquitous slave trade on the west coast of Africa; slavery invariably passing towards extinction, with a diversification of industries, no matter how humble and small the development of those industries.

There is scarcely another industry which can show as many improvements, inventions, and novelties during the last ten years as the manufacture of soap, and yet, though there is no lack of good literature, there is not a book in the English language which treats the subject so fully as to answer all the demands and requirements of modern times.

To supply this want is the object aimed at in the preparation of this volume. It is chiefly based upon three recent German works, viz., *Handbuch der Seifenfabrikation*, edited by Dr. C. Deite; *Handbuch der practischen Seifenfabrikation*, by Alwin Engelhardt, and *Die Technologie der Fette und Oele*, by Dr. Karl Schaedler. These works are the results of many years of prac-




tical experience, and are by men of acknowledged authority. The volume edited by Dr. Deite consists of contributions by various expert soap-boilers, giving each the method of fabrication in a special branch of the industry. A. Engelhardt's work contains many processes of manufacture and formulas which, being the result of years of active labor as manager and proprietor of large soap-works, and having been thoroughly tested, can be confidently recommended to every soap-boiler. To Dr. Schaedler's work the editor of this volume is indebted for much of the matter relating to the raw materials, the preparation of fatty acids, and the manufacture of candles.

In order to make the present treatise as complete and reliable as possible, much time and care have been devoted to the gathering of information from all available and widely-scattered sources; and the editor takes pleasure in acknowledging his indebtedness to the work on the same subject by R. S. Christiani, published by the publishers of the present volume; to the writings of W. L. Carpenter, A. Watt, C. R. A. Wright, C. H. Schmidt, and many others; also to Dr. William H. Seaman, of the United States Patent Office, for Lists of Patents relating to the subjects treated of in the volume.

WILLIAM T. BRANNT.

PHILADELPHIA, May 5, 1888.





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A

# PRACTICAL TREATISE

ON THE

## MANUFACTURE OF SOAP AND CANDLES.

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### PART I.

#### MANUFACTURE OF SOAP.

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#### CHAPTER I.

##### HISTORY OF THE MANUFACTURE OF SOAP.

IF we inquire into the origin of the manufacture of soap, we find that a detergent corresponding to our soap is not mentioned by any writer before the Christian era. It is frequently asserted that soap was known to the authors of the Old Testament; but the Hebrew words used in the passages\* are stated by authorities to refer to vegetable and mineral lyes, *i. e.*, potash and soda in some form.

In Homer's time the cleansing of clothes seems to have been effected by simply rubbing or pounding in water without any addition, for he tells how Nausicaa and her attendants washed clothes by stamping them with their feet in pits filled with water.

Later on the juices of certain plants were employed as detergents and also natural soda and wood ashes, and the fact that the strength of alkalies can be increased by lime was already known to Paulus Ægina. Fuller's earth was, however, the principal agent used for washing in ancient times, the fuller's art being due, it appears, to one Nicias, the son of Hermais. The Roman

\* Jeremiah ii. 22, and Malachi iii. 2.

fullers (*fullones*), who washed dirty garments, were persons of no little importance. Their trade and the manner of carrying it on were regulated by laws. At one time, fuller's earth, found of a superior quality in Staffordshire, Bedfordshire, and other counties of England, was considered so indispensable for the dressing of cloth that, to prevent foreigners from rivalling English fabrics, it was made a contraband commodity, and its exportation made equally criminal with the heinous and wicked export of wool.

The elder Pliny gives us the earliest account of soap as having been first manufactured by the Gauls, and used by them as a cosmetic, and for dyeing the hair red. He also states that it was made from tallow and ashes, the best being prepared from goat's suet and beechwood ashes, and that the Gauls employed it both in a solid and liquid form.

From this statement by Pliny it has been generally concluded that the invention of soap was due either to the Gauls or the Germans. E. Moride,\* however, contests the correctness of this conclusion. He is of the opinion that Pliny's statement simply refers to the application of soap as a cosmetic and hair-dye, and believes the Phoenicians, who settled in Gaul 600 B. C., to have been the actual inventors of soap.

It cannot, however, be supposed that the first soap was an artificial product like that of the present day. It was very likely a mixture of oil and wood ash, which was used as a salve in eruptions of the skin and similar diseases. Later on it may have been accidentally discovered that a more effective salve was obtained by mixing the ash with water and burnt lime before combining it with the oil. Thus, no doubt, products were gradually obtained which resembled the present soaps of Algiers, of which Leon Droux writes :† “ In the interior of Algiers the Cabyles bring to market a mass which serves the double purpose of a remedy and for household use. It is a soap prepared almost in the cold way, of a slightly yellowish color, somewhat transparent, and of a jelly-like consistency, but with a very small content of water. It is made from olive-oil and lye, the latter being prepared by

\* Les Corps gras industrielles.

† Les Produits Chimiques, Paris, 1878, p. 186.

allowing water to percolate through a mixture of wood ashes and burnt lime. The Arabs use the salve-like product thus obtained for affections of the skin, as well as for household purposes, and washing wool to be worked into tissues."

As a detergent, soap is first mentioned by the authors of the second century after Christ. The celebrated physician, Galenus, speaks of it as a detergent, as well as a medicament, and considers the German soap as the best, and the Gallic as the next best.

But little is known of the further gradual development of the soap industry. Marseilles, it is said, carried on a considerable trade in soap as far back as the ninth century. In the fifteenth century Venice was the principal market, but was outstripped, in the seventeenth century, by Savona,\* Genoa, and Marseilles. At the same time the manufacture of soap seems to have been carried on to a considerable extent in England, where, in 1622, a company obtained a monopoly for the manufacture of soap, paying annually a tax of £20,000 for 3000 tons of soap. As some of the soap-boilers did not join the company, many disputes arose in consequence of this grant, until the king issued an order that no soap should be sold except it was examined by the company. In 1633 sixteen soap-boilers were summoned before the Star-Chamber for disobedience of this order and infringement of the grant. The accused were condemned to pay a fine varying from £500 to £1500, and to be imprisoned at the king's pleasure. This judgment was executed, two of the soap-boilers dying in prison while the others were liberated after forty weeks. In 1635 the holders of the monopoly offering to pay £2 more tax per ton, their privileges were increased, and a few more soap-boilers, disobeying the mandate, imprisoned. In 1637 the patent was finally bought from the patentees for £40,000, and the factory buildings for £3000. The materials the London soap-boilers had to purchase for £20,000 before they were allowed to carry on their trade.

In France the monopoly system was also in force in the seventeenth century. In 1666 Pierre Rigat, a merchant of Lyons, made a proposition to the king to manufacture, by special methods,

\* Whence the French name of soap (*savon*).



sufficient soap for the consumption of France without importing any of the materials required in the manufacture. Louis XIV. accepted the proposition and gave him the sole privilege for 20 years of erecting factories for the manufacture of white, marbled, and all other kinds of soap, in any place it suited him. The six or seven factories then in existence were allowed to remain, but under the condition that they should not increase their capacity, and would sell their products to Rigat at a fixed price. This patent, being the cause of many disputes, was revoked in 1669.

Many complaints about the adulteration of soap caused the French government, in 1688, to issue special directions for the manufacture. It was decreed (1) that all soap-makers, no matter what kind of soap they produced, must stop the manufacture during the months of June, July, and August of every year; (2) that new oils should not be used in the manufacture of soap before the 1st of May of every year; and (3) that besides barilla, soda, ash, and olive-oil, no other fats or materials should be employed. This decree was partially revoked in 1754, and the manufacturers were allowed to work during the entire summer. Six years later the factories were again ordered to close during the summer, though this time the decree was issued on the recommendation of the soap-boilers themselves. The Revolution of 1789 did away with all these useless restrictions.

But little is known about the soap industry in Germany during the early centuries. The business was carried on on a small scale, and this could not well be otherwise, since, with the impure raw materials, principally crude tallow and wood ashes, one boiling frequently required as many days as hours at present. The industry was further hindered by the general practice of every household preparing its own soap, which continued up to the introduction of artificial soda and tropical vegetable fats. But since then many large factories have been established, and, as the German soap-boilers applied to the trade its true chemical character, they produced superior goods. The soft soap of Germany is still much used for household purposes as well as for manufacturing, and it has acquired a reputation for excelling in quality that of other countries.

In this country there has been a steady progress in the im-

provements constantly making in this important branch of industry, until now we are producing goods which for quality compare favorably with any made elsewhere; moreover, we have invented much new and improved machinery and apparatus that greatly facilitate the processes, saving labor and time and improving the quality. Thus the United States is at this time but little behind any other country, either in the amount made or in the quality of the article; while in the economy and facility of their manufacture this industry is fully abreast, if not in advance, of that of nearly all other countries, and is steadily progressing, so that it cannot be long before we shall equal in quality and excel in quantity.

Not much advance was made in the manufacture of soap until at the beginning of the present century it commenced to attract the attention of scientific men, and Leblanc gave to the world his splendid process for the production of soda from common salt. But, like many other benefactors of the human race, Leblanc did not reap the reward for his invention. In 1791 a patent for fifteen years was granted to him in France, which was, however, shortly after revoked. The manufactory, which Leblanc had established by the aid of the Duke of Orleans, was just beginning to work when the Revolution put an end to all business; the property of the Duke of Orleans was seized, and the factory being included, the fabrication was stopped. Soon, the Continental war preventing the importation of Spanish sodas, the French industry felt the loss of this important element so essential to its work. Then, on the proposition of Carnot, the Committee of Public Safety obliged Leblanc to sacrifice to the country the fruit of his discovery by making it public. Leblanc was ruined, and after many struggles for redress fell a victim to melancholy, and died, in 1806, by his own hand and in abject poverty.

The next great discovery, and not second in importance, was due to Chevreul, who, by describing the exact constituents of the fatty bodies and making known the processes for their separation, raised soap-making from empiricism and guess-work to its present position as a truly scientific art. By establishing the practical and scientific basis upon which the soap industry is now

carried on, Leblanc and Chevreul may be considered as its founders.

The introduction of new fats and oils, especially of palm-oil, palm-kernel oil, and cocoanut-oil, added an important variety to the lists of soaps, particularly of toilet-soaps. Of great importance, also, was the introduction of caustic soda, which enabled the soap-boiler to prepare with great ease caustic lyes of a high degree.

Soap is one of those products whose capital value, disappearing constantly from circulation, has to be renewed; it is one of the most important products which, after use, are absolutely of no value. With old pieces of glass, window-panes can be purchased, and with rags, clothing; but with soap-water nothing can be done in our households. To be sure, in modern times many attempts have been made to collect the soap-waters from large wash-houses and to separate the fatty acids by sulphuric acid, but this represents but a very small quantity of the fat lost in this way.



## CHAPTER II.

## RAW MATERIALS USED IN THE MANUFACTURE OF SOAP.

THE ordinary soaps of commerce consist of the more or less pure sodium or potassium salts of fatty acids. They are prepared by subjecting fats and fat oils, and also fatty acids, to the action of lyes. The latter are prepared by treating aqueous solutions of alkaline carbonates (soda or potash) with caustic lime, or by simply dissolving the caustic alkalis in water. For some soaps an addition of rosin is also used.

The raw materials used in the manufacture of soap consist, therefore, on the one hand, of fats, fat oils, fatty acids, and rosin, and, on the other, of alkalis. To these must be added, as auxiliary raw materials, water, lime, and common salt.

With these few preliminary remarks we shall proceed to the description of the raw materials, their properties as far as of interest to the soap-manufacturer, and the methods of examining them.

*Fats.*

*Constitution of fats.*—To understand the chemical process which takes place during saponification it will be necessary to consider the physical and chemical properties of fats.

The term “fats” is applied to certain products of the animal and vegetable kingdoms with the following common properties: they feel unctuous to the touch and form oleaginous fluids, either at an ordinary temperature or when heated. Dropped upon paper they cause transparent stains which do not disappear in time nor by the application of heat. In an absolutely pure condition they are all colorless, inodorous, and tasteless, but, in consequence of the presence of foreign substances, the commercial products are generally more or less colored; the solid fats being yellowish, the oils yellow or yellow-green, and the train-oils red

or red-brown. They are lighter than water and insoluble in it, and but slightly soluble in alcohol, but freely in ether, carbon bisulphide, and volatile oils. They commence to boil at from  $572^{\circ}$  to  $600^{\circ}$  F., whereby they are decomposed, forming volatile acids of a disagreeable and irritating odor. At a still greater heat they form a combustible gas which has three times the illuminating power of coal-gas and is of much greater purity.

According to their consistency, the fats are divided into solid fats or tallows, semi-solid fats or butters and lards, fluid fats or oils, and train-oils, the latter term being applied to the fluid fats derived from various marine animals. The solid fats melt readily, most of them becoming fluid below  $212^{\circ}$  F., while the liquid fats, *i. e.*, oils, assume a firm consistency at a low temperature.

The oils, of all fluids, expand the most by heat, and, indeed, in some cases to such a degree as to render it necessary, for the purposes of commerce, to know exactly the extent of the expansion. For every degree of Celsius the volume of olive-oil increases, according to Preisser, 0.83, of rape-oil 0.89, and of train-oil 1.10; so that 1000 measures of olive-oil, at  $32^{\circ}$  F., will become, in summer, at  $68^{\circ}$  F., 1016.6 measures, 1000 of rape-oil 1017.8, and 1000 of train-oil 1020.

By exposure to the air most fats undergo a gradual change. By the absorption of oxygen several of the liquid fats dry up and are converted into a solid transparent substance. Such oils are called "*drying oils*." Others, called "*non-drying oils*," never dry up entirely, but only acquire a greater degree of consistency and turn rancid, the latter being due to the formation of an acid. Many solid fats also become rancid; palm-oil and cocoanut-oil, as found in commerce, being, for instance, frequently in that state of decomposition, though they do not show a bad odor.

While pure, unaltered fats, with the exception of castor-oil, are almost insoluble in cold alcohol, they dissolve readily in it when rancid and show an acid reaction; the latter, when not due to admixed foreign substances, always indicates commencement of rancidity, as in a pure, unaltered state the fats are entirely neutral.

*Chemical constitution.*—According to their elementary composition, the fats consist of carbon, hydrogen, and oxygen as distinguished from the so-called mineral fats and oils, which consist

only of carbon and hydrogen. The following table shows the elementary composition of some of the fats :—

	Carbon. per cent.	Hydrogen. per cent.	Oxygen. per cent.
Cocoa-butter . . . . .	75.20	11.90	12.90
German sesame-oil . . . . .	76.80	11.50	11.70
Lard . . . . .	76.54	11.94	11.26
Linseed-oil . . . . .	78.00	11.00	11.00
Mutton-suet . . . . .	78.10	11.70	9.30
Olive-oil . . . . .	77.21	13.36	9.43
Ox-tallow . . . . .	76.50	11.91	11.59
Poppyseed-oil . . . . .	76.63	11.63	11.74
Rape-oil . . . . .	77.21	13.36	9.43

As will be seen from the above table there is a considerable variation in the elementary composition of the fats, and moreover there can scarcely be any doubt that the fat from the same animal is not always alike in composition. The fats, which occur in nature, are not simply chemical combinations, but mixtures of such.

An important step towards the investigation of the nature of fats was made by Scheele, in 1779, by the discovery, whilst engaged in the preparation of lead plaster, of a sweet body soluble in water. He called it "*principium dulce oleorum*," and it is now generally known as glycerin.

But the principal elucidation of the nature of fats we owe to the labors of Chevreul, which commenced in 1812, and culminated in 1823 in the publication of his celebrated work : "*Recherches sur les Corps gras d'origine animale*." As a basis for his researches he used a soap from lard and potash, and established the fact that the oxygen of the air had no part in the saponification, as was formerly supposed by many chemists ; that fat once saponified is not altered by repeated combination with potash ; that generally the fats are mixtures and by decomposition with sulphuric acid form acids in the same manner as by decomposition with alkalies ; that the fatty acids obtained by saponification and the glycerin amount to from  $4\frac{1}{2}$  to  $5\frac{1}{2}$  per cent. more than the weight of the fat used ; that by the combination of fatty acids with lead oxide water is expelled. From the last observations Chevreul concluded that the fatty acids as well as the glycerin contain water chemi-



cally fixed; he compares the fats with the compound ethers which are decomposed in a similar manner into salt and alcohol with the absorption of water.

Chevreul's researches served as a basis for all further studies of the chemical behavior of the fats. The correctness of his observations was in the main confirmed, and his researches extended by others, especially by Berthelot, who enriched our knowledge by the synthesis of the fats from fatty acids and glycerin, and by Heintz, who improved the methods for the examination of fats.

Nearly all the fats occurring in nature are peculiar combinations of glycerin, a body composed of 3 equivalents of carbon, 8 of hydrogen, and 3 of oxygen, and are, therefore, termed *glycerides*. According to the older chemical view, the fats contained a body called glyceryl-oxide or lipyloxide, combined with 3 equivalents of fatty acids, and glycerin was designated as glyceryl-oxide, or as glyceryl-oxyhydrate. But, according to modern chemistry, glycerin is a so-called triatomic alcohol. If, as stated above, glycerin contains 8 equivalents of hydrogen, 3 of them can be represented by acids. If we designate carbon C, hydrogen H, and oxygen O, glycerin has the formula:  $C_3H_5O_3$ , which can also be written:  $C_3H_5H_3O_3$ , or  $C_3\overset{H}{H}_5O_3$ . The 3 equivalents of hydrogen, written separately in the last two formulas, can be represented by acids. Now if 1 equivalent of hydrogen is replaced by an acid, a so-called monoglyceride is obtained; if 2 equivalents are replaced, a diglyceride; and if all 3 equivalents are replaced, a triglyceride. By designating an acid R,

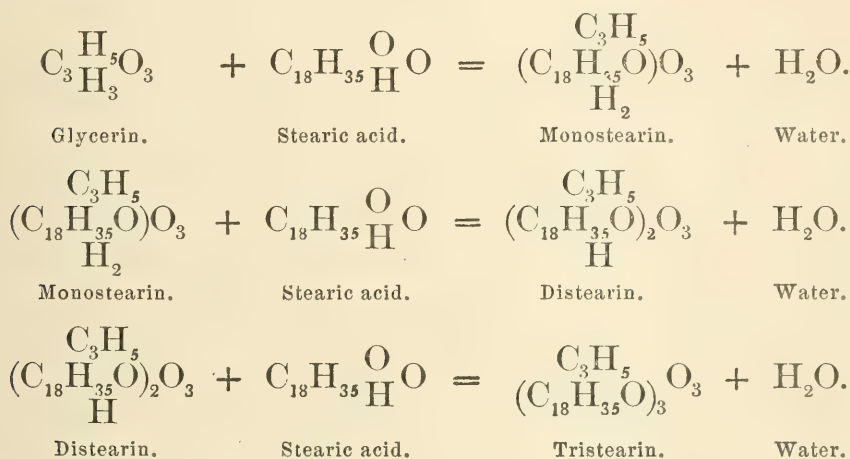
the monoglyceride has the formula  $C_3\overset{H}{H}_2R O_3$ ,

“ diglyceride      “      “       $C_3\overset{H}{HR}_2 O_3$ ,

“ triglyceride      “      “       $C_3\overset{H}{R}_3 O_3$ .

All glycerides occurring in nature, and others thus far examined, are triglycerides, *i. e.*, glycerin in which 3 equivalents of hydrogen are replaced by acids. The acids contained in fats are generally termed fatty acids, the most important being stearic, palmitic, and oleic acids. Artificially the glycerides can be prepared by heating

glycerin and fatty acids together in closed glass tubes. By heating, for instance, equal parts of glycerin and stearic acid in a closed tube to  $392^{\circ}$  F. for thirty-six hours, the monoglyceride, *i. e.*, monostearin, is formed. By heating the monostearin with 3 parts of stearic acid to  $500^{\circ}$  F. for three hours, the diglyceride, *i. e.*, distearin, is obtained, and the triglyceride, *i. e.*, tristearin, by heating monostearin with 15 to 20 times its weight of stearic acid to  $518^{\circ}$  F. The process may be represented by the following equations :—



Many train-oils contain ether-like combinations of another alcohol, cetyl alcohol ( $\text{C}_{16}\text{H}_{34}\text{O}$ ), and wool fat, ether of cholesterin ( $\text{C}_{26}\text{H}_{44}\text{O}$ ), as well as that alcohol itself.

*Fatty acids.*—The fatty acids, which can be separated from the fats, may, according to their composition, be divided into four groups : into acids, 1, of the composition  $\text{C}_n\text{H}_{2n}\text{O}_2$  (usually called acetic series of acids) ; 2, of the composition  $\text{C}_n\text{H}_{2n-2}\text{O}_2$  (oleic acid series) ; 3, of the composition  $\text{C}_n\text{H}_{2n-4}\text{O}_2$  ; and 4, of the composition  $\text{C}_n\text{H}_{2n-2}\text{O}_3$ .

Of the acetic series of acids the following occur in the fats : butyric acid ( $\text{C}_4\text{H}_8\text{O}_2$ ), caproic acid ( $\text{C}_6\text{H}_{12}\text{O}_2$ ), caprylic acid ( $\text{C}_8\text{H}_{16}\text{O}_2$ ), capric acid ( $\text{C}_{10}\text{H}_{20}\text{O}_2$ ), lauric acid ( $\text{C}_{12}\text{H}_{24}\text{O}_2$ ), myristic acid ( $\text{C}_{14}\text{H}_{28}\text{O}_2$ ), palmitic acid ( $\text{C}_{16}\text{H}_{32}\text{O}_2$ ), stearic acid ( $\text{C}_{18}\text{H}_{36}\text{O}_2$ ), arachidic acid ( $\text{C}_{20}\text{H}_{40}\text{O}_2$ ), behenic acid ( $\text{C}_{22}\text{H}_{44}\text{O}_2$ ), cerotic acid ( $\text{C}_{27}\text{H}_{54}\text{O}_2$ ), theobromic acid ( $\text{C}_{64}\text{H}_{128}\text{O}_2$ ). The first four-named acids are known as “volatile fatty acids.” They are liquid at an ordinary temperature, generally oily, and leave grease-stains upon paper which

partially disappear. They are volatile, *i. e.*, they can be distilled without decomposition, and on boiling with water pass over with the aqueous vapor, though their boiling-point is higher than that of water. They are of little importance as regards the technology of fats, as they occur in them in very minute quantities. The other acids, mentioned above, are the actual fatty acids. At an ordinary temperature they are solid, tasteless, and inodorous, cause permanent grease-stains upon paper, and, with the exception of lauric acid, which passes over with ordinary aqueous vapors, can only be volatilized, without suffering decomposition, in a *vacuum*, or with superheated steam. They are insoluble in water, soluble in burning alcohol, from which, however, after cooling, they re-separate in crystals, but freely soluble in ether. Their solutions redden litmus only slightly. When heated they ignite and burn with a bright sooting flame. They are easily melted.

The most important of these fatty acids are lauric, palmitic, and stearic acids, and a knowledge of their properties is required for the examination of the fats.

*Lauric acid* is solid, and when crystallized from dilute alcohol\* forms tufts of white, silky needles. Its specific gravity is 0.883 at 60° F. It melts at 109.5° F. The laurates of the alkali metals are amorphous and soluble in water; the other salts are partially crystallizable, insoluble, or sparingly soluble.

*Palmitic acid* consists of tufts of slender needles. It melts at 143.5° F., and in a fluid state at this temperature has a specific gravity of 0.8527. It solidifies on cooling in a mass of nacreous laminae. The largest portion of it can be distilled without decomposition at about 662° F. It is sparingly soluble in cold alcohol, 100 parts of absolute alcohol dissolving only 9.32 parts. It dissolves abundantly in hot alcohol, so that it can be re-crystallized from the solvent. The alcoholic solution shows an acid reaction. Dilute acids have no effect upon palmitic acid; it dissolves, however, in concentrated sulphuric acid, but on diluting the solution is re-separated without alteration. Boiling concentrated nitric acid attacks it very slowly. The salts of palmitic acid resemble very much those of stearic acid (see below), they being only more sparingly soluble.

\* From strong alcohol lauric acid crystallizes only at 32° F.



*Stearic acid*.—Pure stearic acid crystallized from alcohol consists of nacreous needles or laminae, which melt at  $156^{\circ}$  to  $156.5^{\circ}$  F. to a colorless liquid, which on cooling solidifies to a white, fine, scaly, crystalline mass, lamino-crystalline on the fractured surface. By heating to  $680^{\circ}$  F. it commences to boil, with partial decomposition. With a decreased pressure it distils without alteration. In distilling with superheated steam it apparently also passes over without alteration, but experience in the distillation of fatty acids on a large scale has shown that a small portion is decomposed and passes over into solid hydrocarbons of the series  $C_n H_{2n+2}$ .<sup>\*</sup> At  $11^{\circ}$  C. ( $51.8^{\circ}$  F.) its specific gravity is the same as that of water; at higher temperatures it floats upon water, expanding more quickly by heat than the latter. At low temperatures it is heavier than water, having a specific gravity of 1.01 at  $32^{\circ}$  F. Its specific gravity when melted at  $156.5^{\circ}$  F. is 0.8454. It is tasteless and odorless and not unctuous to the touch. It is insoluble in water, but freely soluble in hot alcohol. In cold alcohol it is even more sparingly soluble than palmitic acid. Forty parts of absolute alcohol dissolve 1 part of stearic acid; in ether it dissolves freely. At  $73.5^{\circ}$  F. 1 part of benzol dissolves 0.22 part of stearic acid, and 1 part of carbon bisulphide 0.3 part.

The salts of stearic acid and the other non-volatile fatty acids are called soaps, the alkaline salts being soluble in water, while the others are insoluble or only sparingly soluble. By boiling stearic acid with aqueous solutions of carbonate of soda or potash, carbonic acid is expelled and stearates are formed. The alkaline salts in their purest state are crystallizable. Their behavior in water is also characteristic of the alkaline salts of other fatty acids. They are sparingly soluble in cold water, but when boiled with not too large a quantity of it they yield a clear solution, which, however, congeals to a turbid, viscid mass. With a large quantity of water they do not yield a clear solution, but a turbid fluid which, on shaking, gives a strong lather which remains standing for some time. The stearates are separated from their solutions by sodium chloride (common salt). The potassium salt can be

<sup>\*</sup> Benedict. *Analyse der Fette und Wachsarten*. Berlin, 1886, S. 11.

converted into the sodium salt by repeated treatment ("salting out") with common salt. The alkaline stearates are freely soluble in alcohol. On cooling the soaps generally separate from concentrated solutions as a jelly, which assumes, however, a crystalline form after long standing. In ether and petroleum ether they are insoluble.

*Potassium stearate* forms crystals with a fatty lustre, which dissolve in 6.6 parts of boiling alcohol. By compounding its hot aqueous solution with much water the acid salt, which is insoluble in water, is precipitated in nacreous scales. *Sodium stearate* is very much like the potassium salt, and consists of nacreous laminae. The aqueous solution of *ammonium stearate* yields, on heating, ammonia, and is converted into the acid salt. The other salts of stearic acid can be obtained by precipitating aqueous solutions of sodium stearate or alcoholic solutions of stearic acid with the acetates of the respective metals. The stearates of calcium, strontium, and barium form crystalline precipitates. The magnesium salt is also crystalline; it is almost insoluble in cold alcohol, but sufficiently soluble in hot alcohol to allow of its re-crystallization from it. The salts of the heavy metals are mostly amorphous, the lead salt being fusible without decomposition at 257° F.

For the quantitative determination of stearic and palmitic acids, it is of importance to know that their insoluble salts are partially decomposed by washing. By washing, for instance, the barium salt, the barium passes into the solution, the residue containing free fatty acid, which can be extracted with alcohol. For a closer examination the fatty acids must, therefore, never be weighed in the form of their salts, but first be liberated from them.

Chevreul distinguished, among the fatty acids obtained by the saponification of fats, one which he termed margaric acid, which, however, has been shown by Heintz to be a mixture of stearic and palmitic acids.

For the manufacture of stearin candles the behavior of stearic and palmitic acids, when melted together, is of great importance. Heintz found that mixtures of fatty acids are analogous to many alloys of metals in showing a lower melting-point than their separate components. The following table shows the behavior of mixtures of stearic and palmitic acids:—

Melting-point. Degrees F.	Composition of mixtures.		Manner of congealing.
	Stearic acid.	Palmitic acid.	
153.0°	90	10	Crystalline scales.
149.5	80	20	Slender crystalline needles.
145.0	70	30	“ “ “
140.0	10	90	Beautiful large needles.
135.5	20	80	Very indistinctly needly.
134.0	50	50	Large-foliated crystalline.
133.0	40	60	“ “ “
132.0	35	65	Non-crystalline, lustrous.
131.0	32.5	67.5	“ “ “
131.0	30	70	“ “ lustreless.

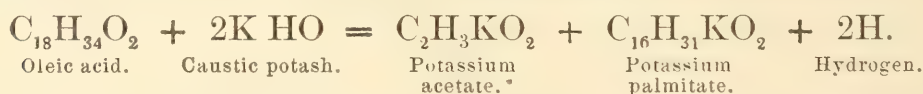
Though the lowering of the melting-points decreases the value of the mixtures for the manufacture of candles, this disadvantage is more than counterbalanced by the importance of the other alterations the fatty acids undergo by being melted together. The pure acids are soft, of a loose structure, and very friable. By mixing them together they acquire sufficient density and hardness, so that they can be subjected to the pressure required to expel the oleic acid. The pure acids in congealing contract to such a degree that candles prepared from them have an unsightly appearance. A mixture of the acids being but slightly crystalline to amorphous, candles manufactured from the semi-congealed mass are dense and non-crystalline. Candles from pure fatty acids are soft, opaque, friable, and lustreless; candles from a mixture, hard, lustrous, and transparent.

Of the oleic acid series the following occur in the fats: tiglic acid ( $C_5H_8O_2$ ), hypogaeic, or physetoleic acid ( $C_{16}H_{30}O_2$ ), oleic acid ( $C_{18}H_{34}O_2$ ), doeglic acid ( $C_{19}H_{36}O_2$ ), and erucic acid ( $C_{22}H_{42}O_2$ ). At an ordinary temperature these acids are partially solid and partially liquid, but they all melt at a somewhat increased temperature. In regard to the proportions of their combinations, they are similar to the acids of the first series.

The most important of the second series is oleic acid. In a pure state it is liquid at an ordinary temperature; it congeals at 39° F. to a hard, crystalline mass, which melts at 57° F. Its specific gravity is 0.898 at 50° F. In an entirely pure state it is a colorless, somewhat thickish, substance, without taste or smell,



and does not redden litmus paper. Oleic acid, in the solid state, oxidizes but slowly in the air; but when melted it rapidly absorbs oxygen, becomes yellow, and acquires a rancid taste and odor, and a decidedly acid reaction. It is not absolutely insoluble in water, very soluble in cold alcohol, and dissolves in all proportions in ether. By the addition of large quantities of water the greater portion of the acid re-separates from its solutions. It volatilizes with overheated steam, or *in vacuo*, without decomposition, but by itself it cannot be distilled at an ordinary pressure. Nitrous acid converts it into the isomeric elaidic acid, a soft, opaque, crystalline mass, without lustre, melting at  $111^{\circ}$  to  $113^{\circ}$  F. It is soluble in alcohol and ether, insoluble in water, and shows an acid reaction. At a temperature of  $140^{\circ}$  F. it absorbs oxygen, whereby it loses its power of congealing. Oxidized oleic acid is not converted into elaidic acid. When heated with caustic potash oleic acid is resolved into palmitic acid and acetic acid:—



The behavior of the salts of oleic acid, as regards their solubility in water, is similar to that of the salts of solid fatty acids, the alkaline salts alone being soluble; all the other salts are, however, soluble in alcohol, and some also in ether, the lead salt belonging to the latter. The alkaline salts separate from their aqueous solutions by the addition of an excess of alkali, sodium chloride, etc. All the salts of oleic acid are softer than those of the solid fatty acids, and mostly fusible without decomposition.

*Sodium oleate* ( $\text{C}_{18}\text{H}_{33}\text{NaO}_2$ ) can be obtained by crystallization from absolute alcohol. It dissolves in 10 parts of water at  $53.5^{\circ}$  F., in 20.6 parts of alcohol of 0.821 specific gravity at  $55^{\circ}$  F., and in 100 parts of boiling ether. The *potassium salt* forms a transparent jelly by far more freely soluble in water, alcohol, and ether than the sodium salt. The *barium salt* is a crystalline powder insoluble in water, and at  $212^{\circ}$  F. cakes without melting. By boiling alcohol it is taken up with difficulty. *Lead oleate* is a loose, white powder, melting at  $176^{\circ}$  F. to a yellowish fluid; after cooling it is, however, rigid and brittle, and remains transparent.

Only two acids of the third group have thus far been found in the fats, viz., linoleic acid ( $C_{18}H_{32}O_2$ ) and elaeomargaric acid ( $C_{17}H_{30}O_2$ ). Of these linoleic acid alone is of any importance, its glyceride forming the principal constituent of linseed oil and very likely of other drying oils. It is a slightly yellowish oil, with a specific gravity of 0.9206 at 57° F., and does not congeal in the cold. It has a slightly acid reaction and is readily soluble in alcohol and ether. By nitrous acid it is not converted into elaidic acid, like the actual oleic acid, but becomes only reddish and thickly fluid. It absorbs oxygen from the air more rapidly than oleic acid, and when exposed, in thin layers, to the action of the air, is first converted into a solid, resinous substance, called oxyoleic acid, and finally into a neutral body insoluble in ether.

The barium and calcium salts of linoleic acid are soluble in boiling alcohol, and the calcium, zinc, copper, and lead salts also in ether. Most of these salts are amorphous; the zinc combination alone can be obtained crystallized.

Of the fourth group only one acid has thus far been found in the fats, viz., ricinoleic acid ( $C_{18}H_{34}O_3$ ). At 59° F. it is a thick oil with a specific gravity of 0.940, which congeals on cooling to from 20.75° to 14° F., and is miscible in all proportions with alcohol and ether. It cannot be volatilized without decomposition. It does not absorb oxygen from the air; by nitrous acid it is converted into a solid modification, *ricinelaïdic acid*, which melts at 122° F. Most of the salts of ricinoleic acid can be obtained in a crystalline form, and, as regards their proportions of solubility, resemble those of oleic acid. The lead salt is soluble in ether and fuses at 212° F.

In the examination of fats, titrations of the free fatty acids are frequently executed. The best indicator for the purpose is phenolphthalein. The solution is prepared by dissolving 0.5 to 1 gramme of phenolphthalein in 1 liter of alcohol. It has a yellowish color, and is reddened by the slightest addition of alkali in consequence of the formation of the corresponding salts. These salts are completely decomposed by weak acids, so that fatty acids in alcoholic solution can be sharply titrated with phenolphthalein.

*Alcohols of the fatty group.*—As previously mentioned, three

alcohols take part in the constitution of fats : *glycerin*, *cetyl alcohol*, and *cholesterin*, the most important being *glycerin*.

Pure *glycerin* ( $C_3H_8O_3$ ) is a neutral, colorless, and inodorous liquid of the consistency of syrup, having a specific gravity of 1.27 at  $59^\circ$  F. It can be mixed with water and alcohol, and possesses a very sweet taste. Exposed to the air it absorbs as much as 50 per cent. of water. By evaporating an aqueous solution of *glycerin* in a water-bath some *glycerin* volatilizes with the aqueous vapor; the residue cannot be obtained anhydrous even by drying at  $212^\circ$  F. On exposure to heat *glycerin* volatilizes in part, becomes dark, and decomposes, giving off, among other products, a body characteristic of all glycerides, *acrolein* ( $C_3H_4O$ ), the vapors of which are very irritating, attacking most violently the mucous membranes of the nose and eyes. In a current of superheated steam *glycerin* distils without alteration. It possesses great solvent power, dissolving the alkalies, alkaline earths, lead oxide, and salts, especially those which are known to deliquesce in the air. With sulphuric acid it combines to a bibasic acid, which is, however, very deliquescent and cannot be evaporated without decomposition even *in vacuo*; with other acids it forms similar combinations. It was formerly supposed that *glycerin* could not be crystallized, but, when quite pure and anhydrous, it crystallizes on exposure to a very low temperature, especially when agitated, as in railway transport. The crystals are monoclinic, perfectly colorless, and melt at  $60^\circ$  F.

*Cetyl alcohol* ( $C_{16}H_{34}O$ ) is a white crystalline mass, melting at  $122^\circ$  F. and boiling at  $651^\circ$  F. It is insoluble in water, but soluble in alcohol, and very freely so in ether and benzine.

*Cholesterin* ( $C_{26}H_{44}O$ ) crystallizes from chloroform in anhydrous needles of 1.067 specific gravity, which melt at from  $293^\circ$  to  $294^\circ$  F. *Cholesterin* is insoluble in water, very sparingly soluble in cold diluted alcohol, but freely so in ether, carbon bisulphide, chloroform, and petroleum. Its solutions turn the plane of polarization to the left. By careful heating it volatilizes without decomposition; it is, however, best to distil it under decreased pressure.

*Glycerides*.—The most widely distributed, and, therefore, most important glycerides are *tristearin*, *tripalmitin*, and *triolein*, most



fats occurring in nature being mixtures of them. Of further special importance to the soap-manufacturer is the glyceride of lauric acid, or trilaurin, or laurostearin as it is usually called.

*Tristearin*  $\left[ \begin{smallmatrix} \text{C}_3\text{H}_5 \\ (\text{C}_{18}\text{H}_{35}\text{O})_3 \end{smallmatrix} \text{O}_3 \right]$  or, as it is briefly called, stearin, forms colorless, nacreous scales, which melt at  $161^\circ \text{ F.}$ , and congeal at  $158^\circ \text{ F.}$  to an indistinctly crystalline mass. By heating stearin, however, to at  $7^\circ$  above its melting-point, it congeals at  $125.5^\circ \text{ F.}$  to a waxy mass and then melts at  $131^\circ \text{ F.}$  But by again heating it a few degrees above the latter melting-point it reassumes its original melting-point at  $161^\circ \text{ F.}$  Stearin is insoluble in water, sparingly soluble in cold alcohol and ether, but freely in warm ether and boiling alcohol. It is the preponderating constituent of the tallows.

*Tripalmitin*  $\left[ \begin{smallmatrix} \text{C}_3\text{H}_5 \\ (\text{C}_{16}\text{H}_{31}\text{O})_3 \end{smallmatrix} \text{O}_3 \right]$  or, as it is briefly called, palmitin, is contained in a preponderating quantity in the lards. It is best obtained pure from palm-oil. It consists of small, nacreous crystals, very sparingly soluble in cold alcohol, and somewhat more freely in boiling alcohol; on cooling it re-separates, however, in flakes. In boiling ether it is soluble in all proportions. When heated its behavior is very remarkable; it melts at  $123^\circ \text{ F.}$ , but on further heating re-congeals, and then only melts again at  $152^\circ \text{ F.}$

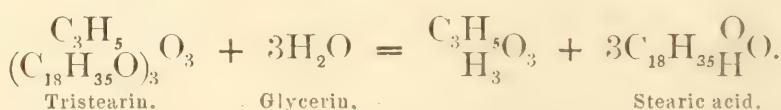
*Laurin* or *laurostearin*  $\left[ \begin{smallmatrix} \text{C}_3\text{H}_5 \\ (\text{C}_{12}\text{H}_{23}\text{O})_3 \end{smallmatrix} \text{O}_3 \right]$  forms slender, white needles grouped in the form of a star or tree, which melt at from  $111^\circ$  to  $115^\circ \text{ F.}$ , and congeal at  $73.5^\circ \text{ F.}$  They are insoluble in water, very sparingly soluble in cold alcohol, but more readily in boiling alcohol, and freely in ether. It is readily saponified by potash lye, forming a clear soap paste. When heated above its melting-point it is decomposed into acrolein and a solid body crystallizable from ether and alcohol.

*Triolein*  $\left[ \begin{smallmatrix} \text{C}_3\text{H}_5 \\ (\text{C}_{18}\text{H}_{33}\text{O})_3 \end{smallmatrix} \text{O}_3 \right]$  or, as it is briefly called, olein, forms the principal constituent of non-drying oils. Pure it is a colorless and inodorous oil which crystallizes in needles at  $23^\circ \text{ F.}$  It is insoluble in water, dissolves with difficulty in cold alcohol,

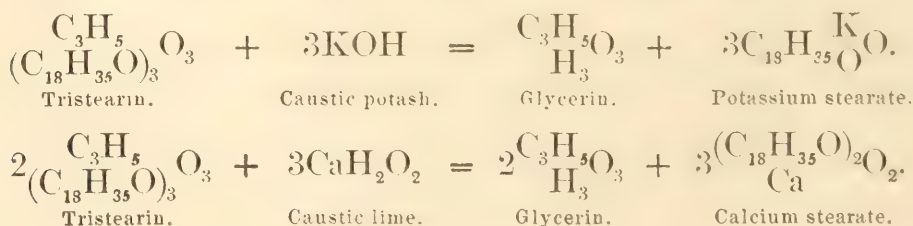
but more freely in hot alcohol, and is miscible in all proportions with ether. Exposed to the air it darkens and becomes acid and rancid by the oleic acid gradually undergoing decomposition. Nitrous acid converts olein into elaïdin which is isomeric with it. This crystallizes in laminae which fuse, according to Meyer, at  $89.5^{\circ}$  F., and, according to Duffy, at  $100.5^{\circ}$  F., and are almost insoluble in alcohol, but freely soluble in ether.

As regards the proportional quantities of stearin, palmitin, and olein occurring in the fats, we would remark, that a fat is the more solid the more it contains of the first two, and the softer the greater the preponderance of the last.

*Saponification of fats.*—By saponification was originally understood only the chemical process which takes place in boiling fats with strong bases whereby glycerin and fat acid salts are formed; but at the present time the term is applied to every reaction by which fats (even without the co-operation of bases) are resolved into glycerin and fatty acids. To understand the process of saponification we must consider that all glycerides are a variety of ethers, and as such possess the power of splitting by the absorption of water into their generators, *i. e.*, into glycerin and an acid. Therefore, for instance,



This splitting of the glycerides is called saponification. It already takes place with water alone, but only at a comparatively high temperature. It is facilitated by adding to the water a small quantity of a base or of an acid; but it is most readily accomplished by using together with the water a sufficient quantity of a base (potash, soda, or lime) to fix the acids formed, whereby the salts of these acids result, which are familiarly known as “soaps.” The process is represented by the following equations:—



The splitting of the glycerides can further be effected by sulphuric acid, whereby first glycerinsulphuric acid is formed and perhaps other sulphuric acid combinations of the acids contained in the fats (sulphostearic acid, sulpholeic acid, etc.).

We would here remark that ammonia, the behavior of which is otherwise analogous to that of the alkalies, does not act in the same manner upon the fats. By shaking a fat oil with liquid ammonia an emulsion is formed; by exposing this to the air the ammonia volatilizes in a short time, and the oil separates without alteration. The oil is also obtained unaltered when a quantity of dilute acid corresponding to that of ammonia is added to the mixture. This proves that ammonia and fat do not form combinations by a simple mixture. But, by allowing ammonia to act upon fats in closed vessels, chemical combinations result, ammonia soap and the amide of the fatty acid being formed.

On the splitting of the natural glycerides into fatty acids and glycerin depends their utilization for the manufacture of soap and candles and the fabrication of plasters. By the latter are generally understood the lead oxides of fatty acids, though the term is also applied to combinations of these acids with other heavy metallic oxides obtained by precipitating soap solutions with metallic solutions.

*Manufacture of stearic acid.*—Though Braconnot and Simonis, as early as 1818, applied the separability by pressure of the fats and oils into a liquid and solid constituent—olein and stearin—for the purpose of obtaining the more solid stearin, the first idea of utilizing this property of the fats and oils for industrial purposes occurred to Gay-Lussac and Chevreul, to whom, in 1825, a patent was granted. Their specifications are very interesting, as they embody nearly all the scientific principles applied at the present time to the fabrication of fatty acids, even the saponification by means of acids, which was practically carried into effect only twenty years later. Their process was, however, a failure, being too much like the methods employed by the chemist in the laboratory, and too complicated for technical purposes. Saponification was to be effected by means of alkalies, and the decomposition of the resulting soap by hydrochloric acid.

The manufacture was placed upon a practical and economical



basis by the introduction by de Milly and Motard, in 1831, of the use of lime in place of alkalies for the saponification of fats, and that of sulphuric acid for the decomposition of the lime-soap formed. Stearic acid, as a material for candles, was first used by de Milly in 1831, in Paris, on the Barrière de l'Etoile, and hence the candles were called "*Bougies de l'Etoile*" and, also, "*Milly candles*."

The saponification of fats by means of a high pressure was patented by Runge as early as 1835, but was first introduced into practice by de Milly, who, in 1855, patented an apparatus suitable for the purpose, which is called an "autoclave."

The fact that the neutral fats are decomposed by concentrated sulphuric acid in a similar manner as by caustic alkalies was known to Achard of Berlin in the year 1777. It was scientifically investigated, in 1836, by Frémy, and finally practically applied in 1840 by Gwynne.

The distillation of the fats was proposed by Gay-Lussac and Chevreul as early as 1825, but was not industrially applied until 1841, when Dubrunfaut introduced the process on a large scale. Nearly at the same time Wilson and Gwynne took out patents for a combination of both processes—decomposition with sulphuric acid, and subsequent distillation.

To the researches of Tilghman and Bertholet we owe the interesting and important fact that water at a high temperature (356° F.) and a pressure of 10 to 15 atmospheres is capable of splitting neutral fats into their constituents.

It still remains to be mentioned that when, according to the researches of L. Kraft and Tessié du Motay, a neutral fat is heated with anhydrous chloride of zinc, a complete incorporation of these substances takes place between 302° and 392° F.; and by continuing the heating for some time and washing the materials with warm water, or, better, with water acidulated with hydrochloric acid, there is obtained a fatty matter, which, on being subjected to distillation, yields the corresponding fatty acid, while only a small quantity of acrolein is formed. The chloride of zinc, becoming soluble in the water used for washing, may be recovered by evaporating the fluid. The yield of fatty acids by this process is the same as that obtained by the use of sulphuric

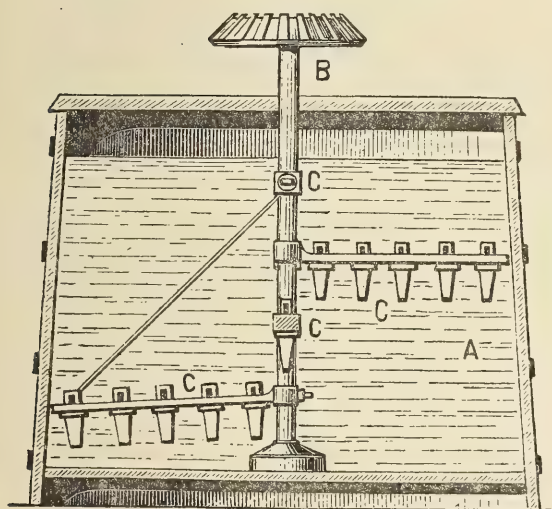
acid, while the fatty acids also agree as to their physical properties. The quantity of chloride of zinc required amounts to 8 to 12 per cent. of the fat.

We will now proceed to give a description of the various methods of saponifying the fats.

1. *Saponification of the fats by means of lime. a, under ordinary atmospheric pressure.* The process upon which the technical separation of stearic acid is based is the same as for soap, and commences with the displacement of the glycerin by means of lime. The resulting lime-soap is then decomposed by sulphuric or hydrochloric acid in order finally to effect the separation of the stearic and palmitic acids by pressure.

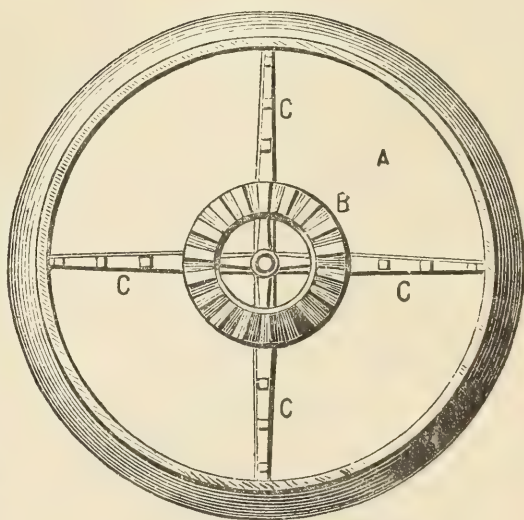
For saponification the tallow or palm-oil, etc., is brought into a vat lined with lead, or into a cemented brick reservoir, the former being about  $5\frac{1}{4}$  feet wide and  $2\frac{1}{4}$  feet deep for 1100 lbs., and the latter having a volume of about 406 cubic feet, which suffices for 4000 lbs. of tallow, 600 lbs. of lime, and 4000 lbs. of water. To prevent the lime-soap from acquiring a yellow color iron vessels and lime, containing iron, have to be avoided.

Fig. 1.



Section.

Fig. 2.



Ground-plan.

*A*, vat; *B*, shaft with bevel wheel; *C*, stirring arms.

The fat, together with 15 per cent. of water, is brought into the vat *A*, Figs. 1 and 2, and steam introduced into the water

through a perforated, convoluted steam-pipe. When the tallow is melted and the water boils, 14 per cent. of pure hydrate of lime, in the form of milk of lime, is added to the fat and mixed with it by means of the stirring arms *C*, set in motion by the shaft *B* with bevel wheel.

Theoretically, 9.12 per cent. of lime would suffice, but 14 per cent. is generally taken, as the excess facilitates saponification, though a larger consumption of sulphuric acid is later on required.

The mixture of lime and fat, at first milky and thin, soon begins to become more thickly fluid by the formation of calcium sebate, and then, gradually, viscid and sticky, so that it has to be stirred; and, in the course of three to four hours, the mass balls together into gray lumps and grains, becoming gradually harder. The introduction of steam and stirring must now be continued until these lumps and grains have completely hardened, show a uniform granular fracture, and can be readily crumbled between the fingers without being unctuous to the touch, which will generally be the case after stirring six to eight hours. To test if saponification is perfect allow a sample to settle in a glass, and, after pouring off the superfluous water, add some alcohol and hydrochloric acid. If saponification be perfect, the separated fatty acid readily dissolves in the alcohol when heated; if not, non-saponified fat in the form of drops of oil floats upon the surface.

When all is at rest and subsided, the lime-soap in the vat forms a gray, crumbly layer floating upon the water containing the glycerin. The water is drawn off through a pipe provided with a strainer and faucet into a large reservoir, and the soap shoveled into the decomposing vat. For the quicker decomposition of the hard, granular lime-soap by the acid it is frequently comminuted by passing through between grooved rollers, the adhering of the soap to the rollers being prevented by a fine jet of water.

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The communication of noxious odors to the neighborhood being unavoidable by the saponification with lime in open vats, it is advisable to use a closed apparatus, the best known being that by



Delapchier. It is a closed, vat-like vessel, provided with mechanical stirrers, and, besides a better and more economical use of the steam, permits of the operation being carried on at a higher temperature up to  $234^{\circ}$  F. The pressure is regulated by safety-valves.

The apparatus constructed by Leon Droux, of Paris, which is provided with a stirring apparatus and intended for saponification under small pressure, also deserves attention. It is in the form of a copper cylinder, generally about 26 feet long, and  $3\frac{1}{2}$  to  $3\frac{3}{4}$  feet in diameter. A shaft running lengthwise through the cylinder is provided with copper stirrers, and makes thirty revolutions per minute. The power required for 6000 lbs. of fatty substance, to which are added 160 lbs. of lime, is scarcely  $\frac{1}{4}$  horse power, the entire result of the operation being 5600 lbs. of fatty acids and 480 lbs. of glycerin of  $28^{\circ}$  B.

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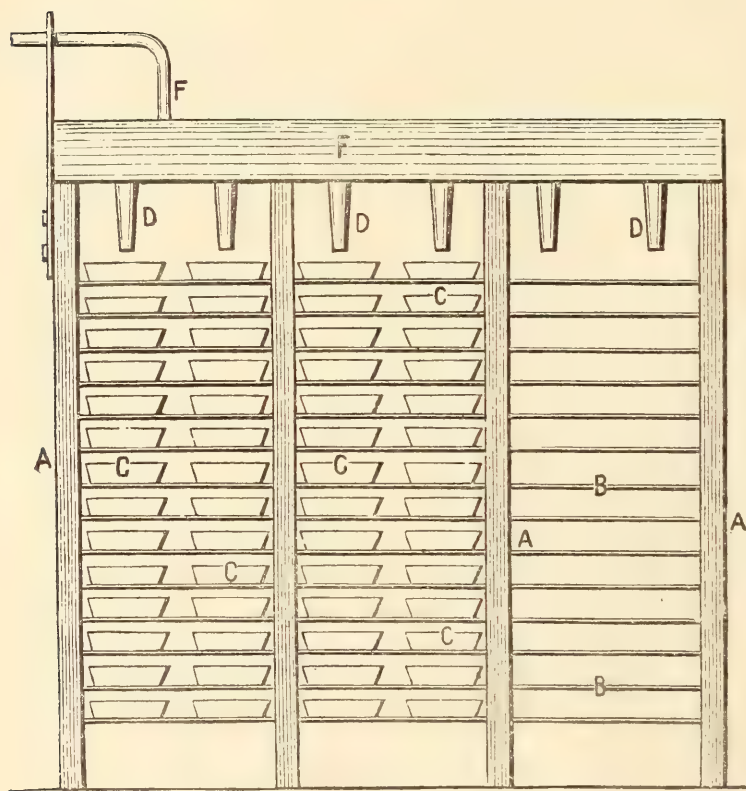
The decomposition of the calcium sebate takes place either in the same vat used for saponification or in a similar vessel also provided on the bottom with a perforated steam-pipe. Decomposition is effected by means of dilute sulphuric acid of  $20^{\circ}$  to  $25^{\circ}$  B., or 1.17 to 1.20 specific gravity, the mass being heated by steam, and stirred for about three hours. The fatty acids having been separated, the introduction of steam is discontinued, and the whole allowed to rest. The melted fatty acids collect on the surface, and the greater portion of the calcium sulphate formed collects as a white crystalline precipitate, while another portion remains dissolved in the free sulphuric acid, and is, therefore, found in the water. In this case, also, the theoretical proportion of sulphuric acid to lime is not used, but always an excess, so that for 100 parts of tallow and 14 parts of hydrate of lime, 24 to 45 parts of English sulphuric acid of  $66^{\circ}$  B. = 1.850 specific gravity are taken.

To completely purify the melted fatty acids from lime, they are pumped or ladled into a vat lined with lead, and thoroughly worked and washed with simultaneous use of steam and dilute sulphuric acid of  $4^{\circ}$  to  $5^{\circ}$  B. (sometimes up to  $12^{\circ}$  B.). After resting for a quarter of an hour, during which the separated

fatty acids are kept fluid, the latter, which are now clear and free from lime, are brought into another large vat, and washed with pure water until no trace of sulphuric acid is found.

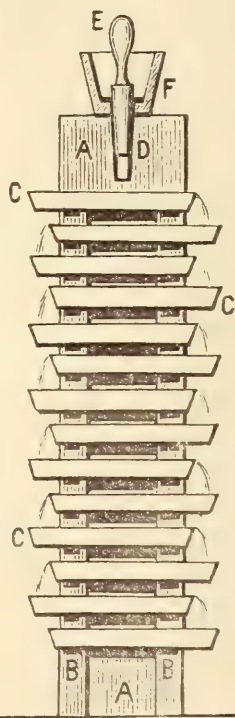
The yield depends on the nature, purity, and treatment of the tallow, and varies between 92 and 95 per cent. of the fat used. It must further be taken into consideration that the large quantity of gypsum separated always envelopes a certain portion of the lime-soap, and carries it to the bottom.

Fig. 3.



Frame for Congealing Fatty Acids.  
Front view.

Fig. 4.



Cross section.

*A*, wooden frame; *B*, iron rods; *C*, trays; *D*, funnel for filling trays; *E*, wooden plug; *F*, pipe from reservoir.

The fatty acids having been thoroughly freed by repeated washing from gypsum, sulphuric acid, etc., are allowed to rest for some time in a melted state, to give the water a chance to completely separate. The fatty acids are then drawn off into trays to congeal or crystallize.

The flat, rectangular trays *C*, Figs. 3 and 4, of heavy tinplate, are provided with a spout, and have each a capacity of  $4\frac{1}{2}$  lbs. of fatty acid, a greater capacity being disadvantageous on account of too slow cooling. The trays are placed upon a wooden framework, *A*, bound by transverse bars of iron, *B*, which support the trays at the same time, in such a manner that the spouts are arranged alternately, and one tray projects above the other. The melted fatty acids are now conducted from the reservoir through the pipe *F* into funnels *D*, which are provided with a wooden plug, *E*, for stopping them. The upper rows of trays being filled, the mass runs over through the spouts into the next row, and so on in zigzag, until all the trays are filled. The wooden plugs *E* are then inserted in the funnels *D*. The trays are kept in a convenient room having a temperature of about  $77^{\circ}$  to  $86^{\circ}$  F. until the fatty acids assume a crystalline form or granulate. At this temperature oleic acid does not solidify, and forms a mother-lye, which contains, besides impurities, a portion of palmitic and stearic acids in solution.

To eliminate the oleic acid, etc., retained between the crystals of the solid mass, and to convert the, at first, smeary and dirty-brown substance into a white, dry, and solid one, the cakes of fatty acid are taken from the trays and subjected, first, to cold and then to warm pressure. This is the more readily accomplished the more crystalline the cakes are. To subject the mass at once to warm pressure would cause loss by stearic acid, etc., passing into the oleic acid.

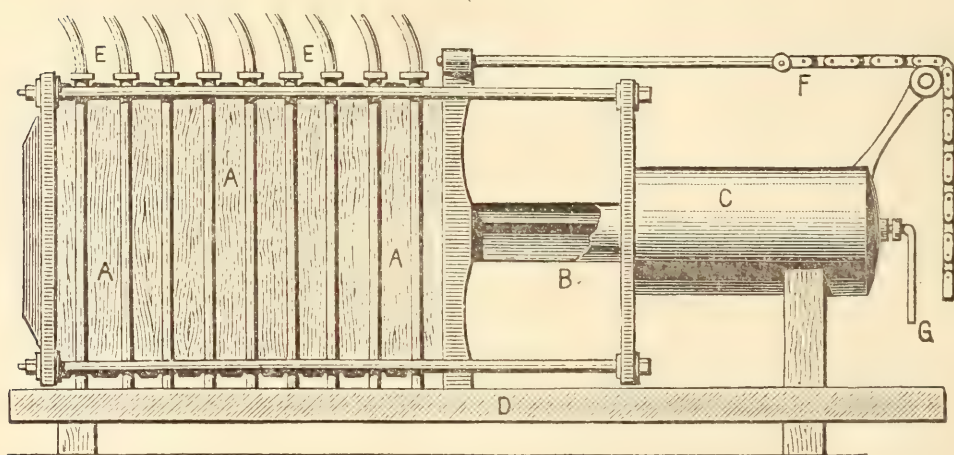
For cold pressing an ordinary hydraulic press is used, the cakes being enveloped in woolen cloths or horse-hair mats. The cakes, taken from the cloths after pressure, are sorted out so that those not completely freed from oleic acid, which is recognized by their dirty color, can be once more subjected to cold, and the rest to warm pressure.

Before subjecting the cakes to the second warm pressure, they are generally thrown into a vat and melted down by blowing in steam for several hours. After settling, the fatty acids are drawn into the previously-mentioned trays and allowed to cool at a temperature of about  $86^{\circ}$  F. When the cakes have assumed a crystalline structure they are cut up into lumps and ground to a mealy



powder by means of a rasping machine, worked usually by steam. This powder is wrapped in woolen cloths, or horse-hair mats, and submitted to warm pressure. A special hydraulic press is required for this purpose, the construction of which has undergone many changes since the introduction of the stearin candle industry. Formerly such presses consisted of a trough, in which the cakes were pressed, a press-cylinder, and cast-iron plates, which, before every operation, were dipped in hot water and placed between the cakes of fatty acid, which, of course, consumed much time. At the present time hollow press plates heated by steam are used. Fig. 5 shows a horizontal hydraulic press with plates which can be heated either by steam or hot water; with the use of the latter the temperature, which is best kept at from  $95^{\circ}$  to  $104^{\circ}$  F., can be better regulated.

Fig. 5.



Horizontal Hydraulic Press.

*A*, cakes to be pressed; *B*, ram; *C*, press cylinder; *D*, collecting reservoir; *E*, steam-pipes; *F*, chain; *G*, stop-valve for press-water.

In the press trough are twenty-four to thirty-six vertical, hollow iron plates, connected with the steam-pipes *E* by means of movable telescoping tubes, and fed from a pipe placed at a higher level. Between these press plates are placed the cakes of fatty acid *A*, enveloped in woolen cloths or horse-hair mats. The temperature and pressure vary with the material operated on, but with good tallow a pressure of 12,000 lbs. per square inch, at a temperature of  $120^{\circ}$  F., is not uncommon. After the pressure is

completed, and the hydraulic ram has passed back, a weight on the chain *F*, which runs over a pulley, unlocks the press, the cakes are taken out, unwrapped, and exposed to the air for a few days to bleach. They are then sorted into three or four qualities, according to the degree of purity. The cakes obtained are hard, dry, and white, except on the places where they have come in contact with iron. The edges always contain some oleic acid and must, therefore, be trimmed off, and the surface of the cakes, if necessary, freed from adhering loose hair from the mats.

The various qualities of solid, fatty acids obtained by hot pressure have to be purified, which is done in lead-lined vats by treatment with dilute sulphuric acid of 3° B. and steam, whereby the fatty acid melts; the same operation is repeated three or four times with pure water until every trace of sulphuric acid is removed. For the complete subsiding of the last water the fatty acid is allowed to rest for some time in a melted state, and is then poured into suitable moulds.

In operating on a large scale 100 parts of crude fatty acid only yield on an average 45.9 per cent. of a mixture of palmitic and stearic acids.

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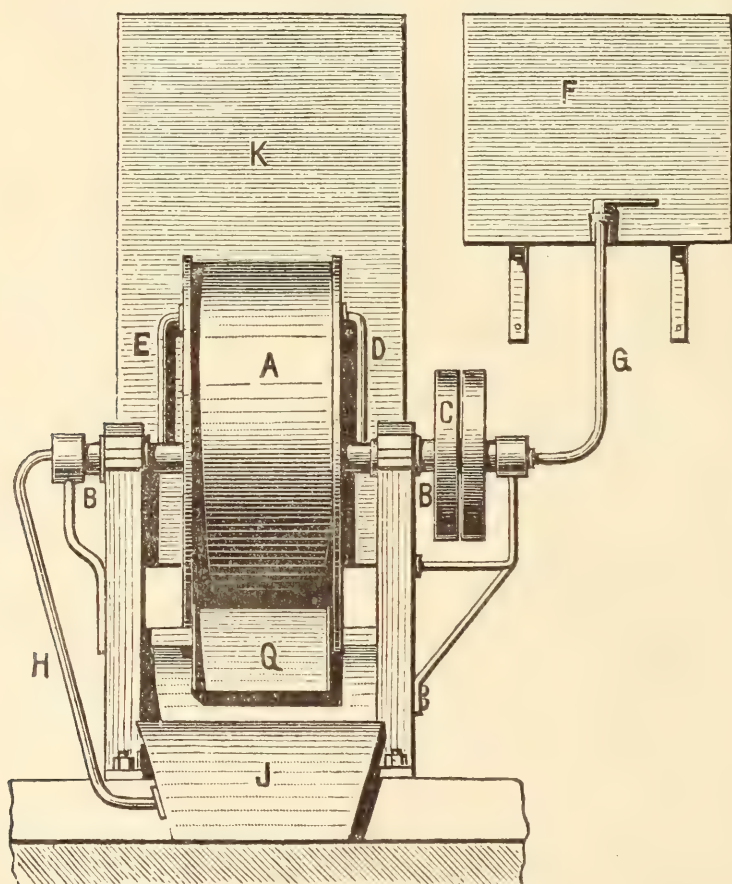
In the stearin manufactories thousands of hundred weights of sulphuric acid are annually converted into worthless gypsum. This would suggest the idea of using barium hydrate for the saponification of tallow and palm oil, which offers the advantage that the sulphuric acid used for decomposing the soap may be utilized at its original cost in the form of barium sulphate (permanent white), and that the water containing the glycerin is separated with greater ease from the heavy barium soap which settles readily than from the lime soap.

The object of a method of saponification with alumina, proposed in 1855 by Cambacères, is also to obtain a more valuable by-product than gypsum. Saponification is not, however, effected by alumina alone, but the fats can be saponified by sodium aluminate, which in this country is used as *Natrona refined saponifier*, an aluminium soap being formed, while sodium oxide is liberated, which can be used for dissolving fresh portions of alumina. In

decomposing the aluminium soap with sulphuric acid, aluminium sulphate is formed, which is used in dyeing, etc., either by itself or after being converted into alum.

In the treatment of fatty acids in the manufacture of stearin, the oleic acid running off during the cold as well as warm pressure, carries away a portion of palmitic acid and stearic acid, and the more, the higher the temperature in the press-room, hence most in summer. La Societé Petit Frères, of Paris, has caused

Fig. 6.



Cooling Apparatus for Stearic Acid Factories. View.

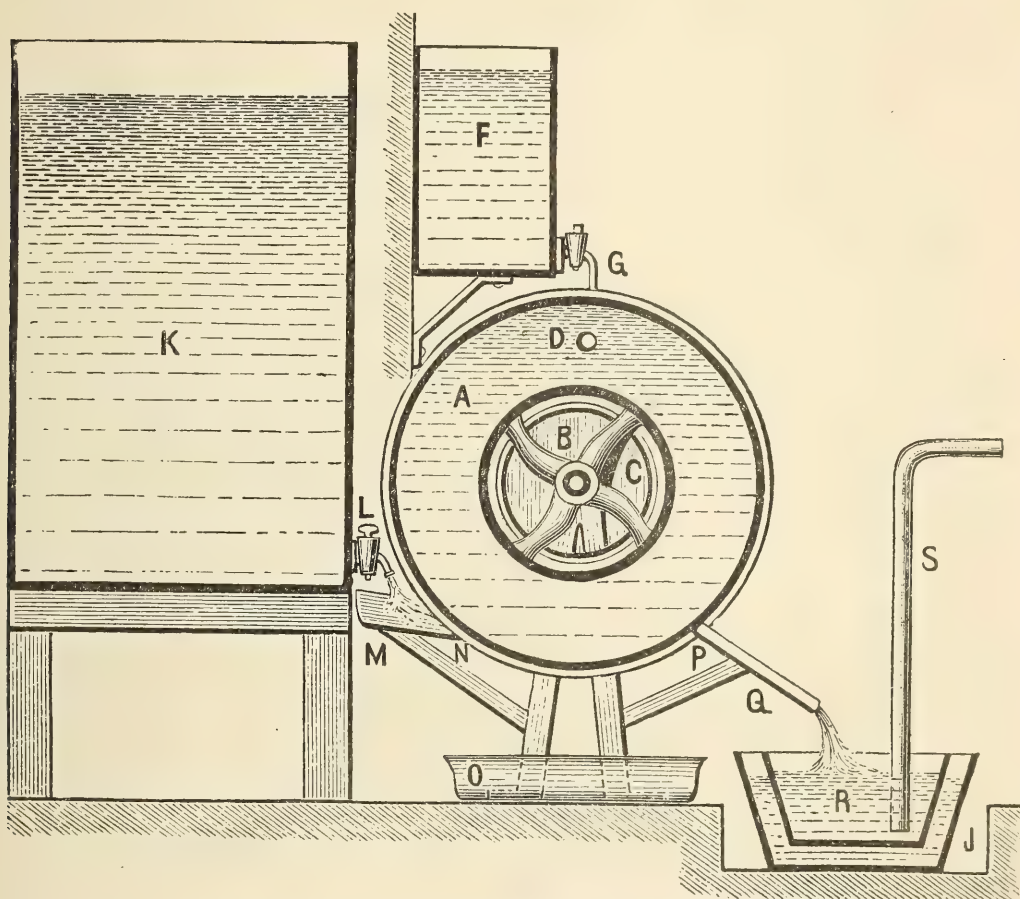
the construction of an apparatus in which the separation of the liquid from the solid mass is executed in a simple and economical manner, and further the mass is converted into thin layers, which of course congeal with greater ease than large volumes.

Figs. 6 and 7 give two views of such an apparatus.



Cold water runs through a drum *A*, which forms an annular closed box. This drum is secured to a shaft *B*, which is revolved by a belt setting the pulley *C* in motion. The shaft *B* is hollow on both ends, the cavities being connected with the sides of the

Fig. 7.



Cooling Apparatus for Stearic Acid Factories. Section.

*A*, drum; *B*, shaft of drum; *C*, pulley; *D*, water conduit; *E*, discharge for water; *F*, water reservoir; *G*, water-pipe; *H*, discharge for water; *J*, water reservoir; *K*, fatty acid reservoir; *L*, faucet; *M*, inclined plate; *N*, strip of rubber; *O*, reservoir; *P*, scraper; *Q*, inclined plane; *R*, fatty acid reservoir; *S*, conduit to filters.

drum by the pipes *D* and *E*. From the reservoir *F*, water is conducted through the pipe *G* to the cavity of the shaft, and therefore through the pipe *D* to the drum *A*, and discharged into the reservoir *J* through the pipes *E* and *H*, thus securing a continual circulation of the water. The fluid fatty acid in the reservoir *K* is now conducted through the faucet *E* upon an inclined plate, *M*, which on the line of contact with the drum is provided

with a strip of rubber, *N*, thus forming a close joint with the drum. Any fatty acid or fat penetrating is caught in the reservoir *O*.

The fatty acid coming through the faucet *L* congeals on coming in contact with the outer wall of the drum, which acts as a cooling surface, and piling up in thin layers upon the drum is removed from it by the scraper *P*, the solid mass falling in the shape of shavings upon the inclined plate *Q*, and from there into the reservoir *R*, which stands in the reservoir *J*. In the reservoir *R* the fatty acid forms a solid mass, being surrounded by cold water in *J*. From the reservoir *R* the mass is then conveyed through the pipe *S* to the filtering press.

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The most advantageous method of freeing stearic acid, etc., from oleic acid still contained in it, is perhaps the one proposed by A. Weise, of Lyons, which consists in enveloping the mass, cooled to  $41^{\circ}$  F., in woolen cloths, and subjecting it to the action of a centrifugal, by which the oleic acid is forced out, while the stearic acid remains in the cloths.

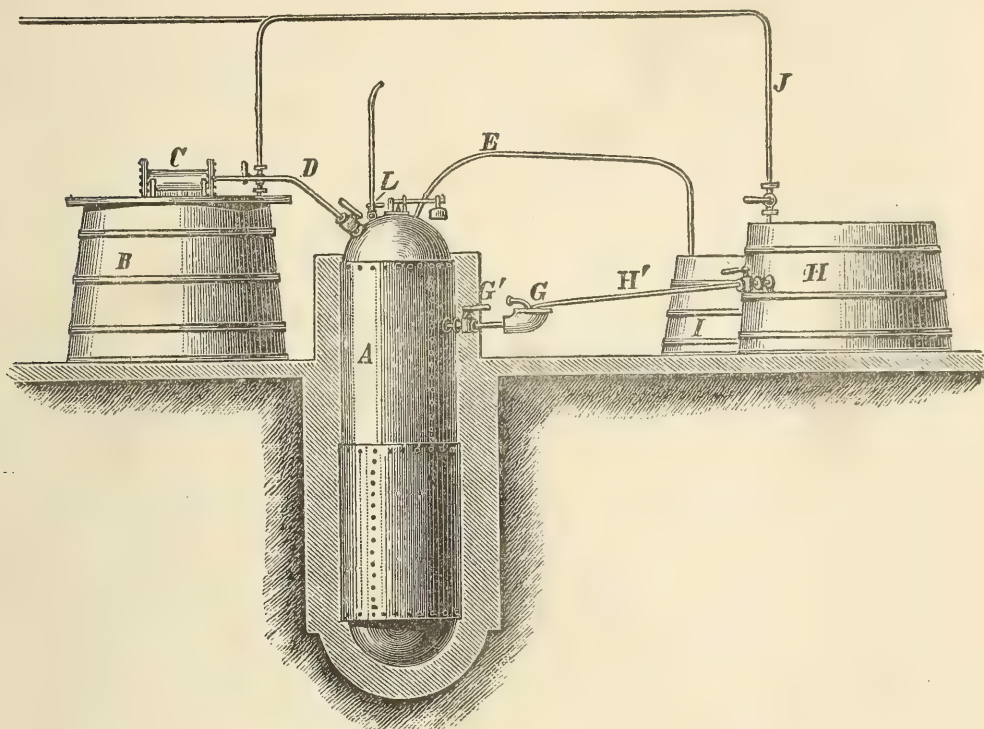
*b. Saponification with little lime, assisted with pressure.*—De Milly has simplified this lime process by using only 2 to 4 per cent. of lime, aided by a high steam pressure of 150 lbs. at a temperature of  $359.5^{\circ}$  F. Fig. 8 shows the apparatus, called an "autoclave," used for the purpose. *A* is a closed cylindrical vessel of copper, 4 feet in diameter and 16 feet high, and provided with a safety-valve, manometer, and manhole. By the pipe *L* it is connected with a boiler, which furnishes steam of a pressure of 8 atmospheres. The steam boiler consists of two heating tubes, placed above each other, and connected by two pipes. These two heating tubes have the same diameter ( $23\frac{1}{2}$  inches) and the same length (16 feet), and are heated by a fireplace.

Steam is introduced into the autoclave through the cock *L* and a pipe extending to the bottom. The pipes *D* and *E*, extending also to the bottom of the autoclave, serve for emptying the apparatus after saponification is finished.\*

\* Instead of two discharge-pipes, only one is generally used at the present time. It is provided outside of the autoclave with a three-way cock, from which connections lead to the glycerin vat and the fatty acid vat.

The operation is carried on as follows: The charge of fat (4000 lbs.) is melted by steam in the wooden vat *H*, and conveyed through the funnel *G* into the apparatus; 220 gallons of milk of lime, containing 120 lbs. of lime, are then added. The appa-

Fig. 8.



De Milly's Autoclave.

ratus being thus charged, the cock *G*, on the funnel, is closed, and the steam-cock *L* opened. The steam enters on the bottom, and, penetrating the milk of lime and fat, effects their mixture. The pressure in the autoclave soon rises to 8 atmospheres. In this manner the steam, which must always have the same pressure, is allowed to enter the apparatus for five or six hours, when it is shut off, and the apparatus allowed to rest for two hours for the water containing the glycerin to settle. The cock *D* is then opened, and, the pressure in the autoclave being still sufficient to force out the mass, the glycerin-lye is first expelled, and passes through a filtering arrangement into the vat *B*. The glycerin having escaped and the fatty mass beginning to appear, the cock *D* is closed, the cock *E* opened, and the mixture of fatty acid and



lime-soap brought into the vat *I*. The mixture being all in the vat, 240 pounds of sulphuric acid of  $66^{\circ}$  B., previously diluted to  $14^{\circ}$  or  $15^{\circ}$  B., are added with constant stirring and simultaneous introduction of steam, the decomposition of the lime-soap being finished in about two hours. The steam is then shut off and the fatty acids allowed to settle, which are then brought into another vat and washed.

This process is, according to Buff, still more advantageous by using a small quantity of sodium oxide in connection with the lime for saponification.

James H. Clinton, of St. Louis, Mo., under date of February 23, 1886, has patented the following process for extracting glycerin from fatty substances: He uses a copper digester of the usual form about 4 feet in diameter by about 16 to 20 feet long. An elongated three-inch zinc bar or slab is suspended, preferably in the centre of the digester, by a copper rack; but this bar may be suspended in other parts of the digester, and more than one bar may be used without departing from the essential features of the process.

The three-inch bar described in this process is of about forty pounds weight, this being about the weight required in a digester of the above-stated dimensions, but the invention is not limited to bars of that weight, as they may vary in weight according to the number of bars, the size of the charge, and the quality of the stock.

After the suspension of the zinc bar in the digester about 6000 pounds of fatty substance (in a digester of the size stated) are then let in, and one per cent. of lime, slaked to the consistency of cream, is added to the charge, except in soap stock, when the lime is dispensed with. Steam is then turned on at about 140 pounds pressure, although the pressure may be varied according to the condition of the charge. The steam preferably enters through a perforated coil at the bottom of the digester until there is about 60 pounds pressure on the charge. The steam is then turned off, while about 2000 pounds of hot water from the boiler at a temperature of about  $300^{\circ}$  F. are forced into the digester. It is an important feature in the process that the charge should be brought under sufficient pressure and sufficiently raised in temperature

before the addition of the water. The steam is then again turned on at about 140 pounds pressure and up to about  $355^{\circ}$  F. of temperature, and kept on for about five or six hours, when the saponification is completed in about half the time it takes in the old lime process. When the saponification is complete, the charge is allowed to settle during about 20 minutes, and the then settled glycerin water is blown off through a blowpipe into an evaporating tank. The water, which is at this time at between  $5^{\circ}$  and  $6^{\circ}$  B. of strength, is evaporated until the residue is at about  $28^{\circ}$  B., the specific gravity of glycerin.

The zinc bar, especially when suspended in the centre of the charge, diffuses its influence throughout the whole of the stock in the digester, avoiding the difficulty experienced by the unequal and only partial diffusion of its influence when the zinc is used in granulated or any other form in which it deposits itself on the bottom of the digester, and consequently misses much of its desired effect on the remote portion of the stock.

The zinc bar lasts for six months with two charges per day. The saponification of the stock is so thoroughly effected by this process that, it is claimed, the market value of the glycerin is one and a half to two cents per pound in advance of other stock previous to refining, being not only purer, but also paler in color and finer in quality. The fatty stock is also said to be much firmer and more waxy in its nature and whiter in color, in consequence of the more complete extraction of the glycerin and the reduction of time that it is under the influence of superheated steam.

The digester in this process keeps clean and free from sediment. Only one per cent. of lime is required, instead of three or more per cent. as in the usual lime process. The fatty stock, even from cotton-seed and other oils, when treated by this process is of a firm and waxy nature, and so pure and light a color that it is found eminently adapted for the production of superior articles of manufacture in many lines.

A very small amount of acid is used to settle the lime in the fatty stock, only  $2\frac{1}{2}$  per cent. instead of  $7\frac{1}{2}$  or more, as in the old system. This process can be used without any change in the usual lime process digester. It is found to be of advantage to have no vent for escaping steam, but to condense the same within

the digester, as there is great loss with steam escaping when under 140 lbs. pressure.

2. *Saponification of the fats with sulphuric acid and subsequent distillation of the fatty acids.*—The so-called “Fremy’s” acids, which comprise the acids known as sulphostearic, sulphopalmitic, sulpholeic, and sulphoglyceric, are formed by gradually adding to olive-oil, lard, etc., half their volume of concentrated sulphuric acid with the avoidance of all heating. The oil, etc., becomes thickly fluid and viscid, acquiring at the same time a slight coloration, the product being the above-named acids, of which the last mentioned is completely soluble in water, and in water containing salt and sulphuric acid, while the first three are only soluble in a small quantity of water, but insoluble in water containing salt and sulphuric acid. If the mixture of acids is compounded with double the volume of water, the pure, fatty acids separate on top, while the water contains the free sulphuric acid and the sulphoglyceric acid; the latter, however, is also split by boiling water.

The above is the basis of the manufacture of stearic acid by means of sulphuric acid. It being scarcely possible in scientific experiments to keep the process within the mentioned limits, they are far exceeded in working on a large scale; the glycerin succumbs almost entirely to the destructive action, and the fatty acids do not completely escape.

England and France both lay claim to the discovery of this process, the latter basing its claim upon the labors of Fremy, Melsens, and Dubrunfaut, and the former upon the patents granted to Jones, Wilson, and Gwynne. In both countries, however, too much sulphuric acid was at first used, and by it remaining too long in contact with the fat, not only the glycerin, but also a portion of the fatty acids, especially of oleic acid, was destroyed, a black, pitchy product of decomposition being formed under the development of sulphuric acid.

At first 60 to 80 per cent. of sulphuric acid was used, which was gradually reduced to 30 per cent., then to 15 per cent., next to from 10 to 12 per cent., and finally to 4 per cent., and even to 3.75 per cent., it being, of course, self-evident that the less sulphuric acid is used, the higher the temperature must be.

The facility with which fats decompose varies very much, tal-



low, for instance, decomposing with difficulty. Decomposition is, however, readily effected with fats in a state of spontaneous decomposition, *i. e.*, of rancidity, either from natural admixtures, which cause them to ferment, or from accidental impurities acting in the same manner. Hence, only such fats are used as, on account of their condition and the impurities they contain, are not suitable for saponification with lime. To this class belong bone-fat, offal from slaughter-houses, kitchens, etc., the products of decomposition by means of sulphuric acid, of the soap water obtained from wool-spinning and cloth-making mills, residues of the refining of fish and other oils, residues of tallow rendering, etc.

The above-mentioned materials being generally very much contaminated, the grosser impurities must first be eliminated by remelting and allowing the mass to subside. They are then brought into a lead-lined kettle, which is provided with a stirring apparatus, and heated by the introduction of steam between a double bottom. A quantity of sulphuric acid, varying according to the nature of the fats—12 per cent. for offal fat, 6 per cent. for palm-oil, or an average of 9 per cent.—is then brought into the kettle, the latter is heated by steam, and the stirring apparatus started. The temperature applied varies generally between  $212^{\circ}$  and  $239^{\circ}$  F., though it is sometimes raised to  $320^{\circ}$  F., and even to  $350^{\circ}$  F. During the heating and stirring the mass swells up and acquires a brown coloration, few volatile fatty acids, but large quantities of sulphurous acid being evolved; the development of the latter is partly due to the action of the sulphuric acid upon the impurities in the fat, and partly to the decomposition of the glycerin-sulphuric acid by the heat, the neutral fat being converted into a mixture of sulpho-fatty acids and sulpho-glyceric acid.

The process of saponification is complete after some fifteen to twenty hours' application of heat and stirring. Two different products are always obtained by the decomposition of the fats: the fatty acids and the products originating from the destruction of the organic substances by the sulphuric acid. With a temperature of  $177^{\circ}$  F. the products of decomposition appear as a soft, elastic body, which dissolves in the fatty acids, but contains not a trace of them as shown by its behavior when treated with alkalies. With a higher temperature, they form a black, tarry

mass more or less hard after cooling, which, however, does not completely separate from the fatty acids on standing, so that the latter always retain a black or brown color, and the tarry mass always absorbs a number of fatty acids, especially oleic acid, which can be extracted with benzene or bisulphide of carbon.

The destroyed and carbonized portion of the fat amounts sometimes to 20 per cent., and generally to 16 per cent.

By the process as conducted by De Milly, the coloration of the fatty acids as well as the destruction of the oleic acid is avoided, and distillation rendered unnecessary for a portion of it. The fat is heated to  $248^{\circ}$  F., and mixed with 6 per cent. of sulphuric acid, but the time of the action of the sulphuric acid is limited to two or three minutes, the mixture after this time being diluted with boiling water. The resulting product is twice pressed, and yields, on the one hand, a white press-cake, which can be used at once, and, on the other, a colored oleic acid, which is subjected to distillation. This process, it is claimed, combines the advantages of two methods, in so far as it gives a greater total yield of material for candles than saponification with lime, and, further, that the non-distilled portion of the fatty acids possesses the good qualities of stearic acid produced in the autoclave.

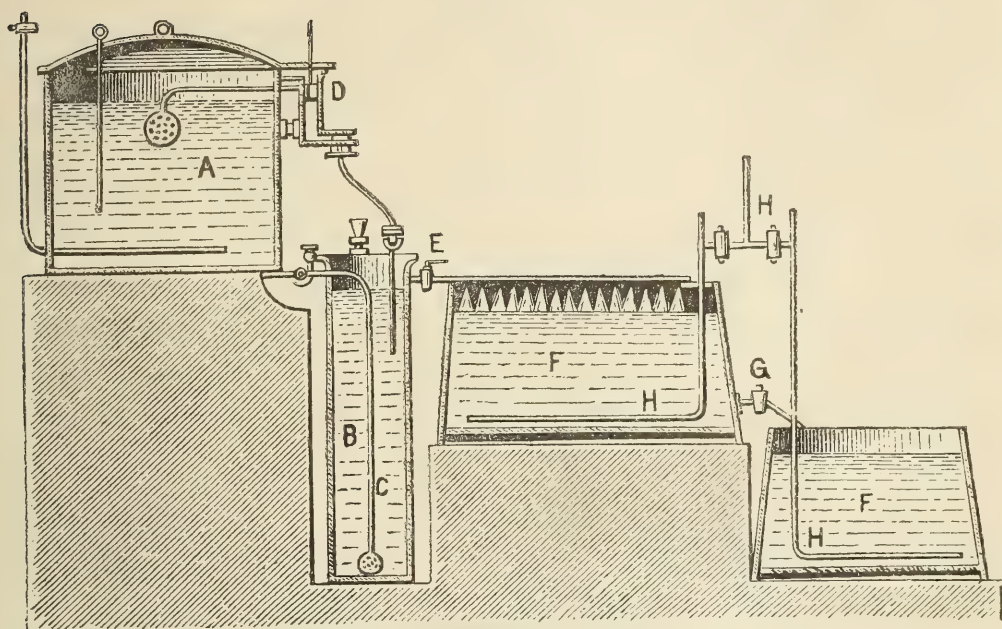
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For a rapid and complete decomposition of the fat with little acid, which need not be concentrated, and hence has not a carbonizing effect, Hughes recommends the following continuously-working apparatus (Fig. 9):—

About 12,000 lbs. of solid fat, to which is added 20 per cent. of oleic acid, are brought into the receptacle *A*, and heated by means of a steam-coil to from  $250^{\circ}$  to  $300^{\circ}$  F., the same temperature being kept up during the entire operation. The heated fat is discharged into the acid vat *B*, which contains 2440 lbs. of sulphuric acid and a few per cent. of nitric acid. If the fatty acids are subsequently to be distilled, sulphuric acid of  $53^{\circ}$  B. and  $220^{\circ}$  F. is used, but only of  $50^{\circ}$  B. if the fatty acid is to serve undistilled for the preparation of colorless fatty acids. The fat rising to the surface collects there, and is reconveyed to the reservoir *A* by means of the pump *D*, and repeatedly makes the

circuit through the acid in the vat *B*, as large a quantity being discharged from *A* as is lifted by the pump from *B*. By this method the points of contact are constantly renewed, and the fat is conveyed in proportionally small quantities through large

Fig. 9.



Hughes's Apparatus for Decomposing Fats.

*A*, fat reservoir ; *B*, acid vat ; *C*, conduit for introducing the fat ; *D*, pump ;  
*E*, discharge cock ; *F*, wash vats ; *G*, discharge cock ; *H*, steam conduit.

quantities of acid. When a sample shows the end of the reaction, the cocks on *A* and *E* are opened, and the fatty mass is discharged into the wash vats *F*, where it is washed for four hours with water acidulated to 12° B. if it is to be pressed without distillation ; otherwise it is drawn off, after resting for two hours, through the cock *G* into the vat *F*, and here washed for three hours with pure, boiling water, which has to be twice renewed, 2 per cent. of common salt being added the last time.

When saponification is complete, the products of the action of sulphuric acid have to be subjected to the so-called "washing," *i. e.*, the sulpho-acids have to be decomposed by water. A com-



plete separation can, however, only be effected at  $212^{\circ}$  F., as in the cold, or even at from  $104^{\circ}$  to  $122^{\circ}$  F., the acid combinations form an emulsion with water, a large quantity of which is readily carried away with the water. After saponification the mass is allowed to cool for three or four hours, and is next transferred to large wooden vats lined with lead and previously filled one-third with water. At the bottom of these vats steam-pipes are fitted, by means of which the fluid contents are soon heated to  $212^{\circ}$  F. By this the sulphuric acid and the fatty acid are dissociated, and these bodies, partly combined with a larger quantity of hydrogen and oxygen than was present in the fatty acids from which they were formed, partly also in an unaltered condition, are found floating on the surface. After having been repeatedly triturated with boiling water, the fatty acids are tapped or poured into a vessel filled with water heated to from  $104^{\circ}$  to  $122^{\circ}$  F. for the purpose of allowing the impurities to become deposited. The clarified fatty acids are then heated in flat pans, in order to evaporate the water, while the separated "glycerin tar" (*glycerine goudroneuse*) is extracted with bisulphide of carbon, to recover the fat contained in it.

That an essential change takes place in the fats is confirmed by the fact that fatty substances thus treated show a higher melting-point, for instance :—

	Kitchen offal.	Palm oil.
In an ordinary state melts at . . . .	$75.5^{\circ}$ F.	$86^{\circ}$ F.
After treatment with acid at . . . .	97	100.5
After washing at . . . .	100.5	111

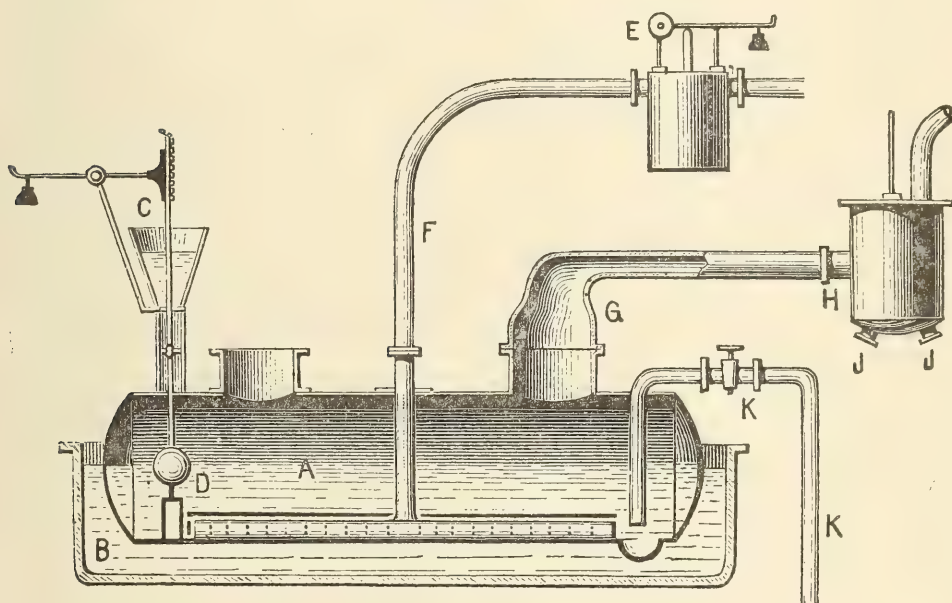
The fatty acids having generally a dark color, distillation is necessary, requiring, however, several precautions. Distillation over an open fire would convert the fatty acids into oil, gas-tar, and a carbonaceous residue, if the heat were sufficiently high. But when the temperature is properly regulated, and the fatty acids are protected from the direct action of the fire, and air is completely excluded from the distilling apparatus, the fatty acids distil over without undergoing any essential change. These conditions are complied with by the use of superheated steam at a tem-

perature between that of the boiling point of water and that of the fatty acids. The temperature of steam at which the separate fatty acids pass over varies very much, that required for palmitic acid being from  $338^{\circ}$  to  $354^{\circ}$  F., for oleic acid  $392^{\circ}$  F., and for stearic acid  $446^{\circ}$  F.

Various kinds of stills are used for the distillation of fatty acids, a few of which will be described, as follows:—

Fig. 10 shows Knab's continuously-working distilling apparatus. Superheated steam is conducted into the interior of the

Fig. 10.



Knab's Distilling Apparatus for Fatty Acids.

*A*, kettle; *B*, pan; *C*, funnel; *D*, float-gauge; *E*, box with safety-valve; *F*, steam conduit; *G*, helmet; *H*, collecting vessel; *J*, discharge cock for fat; *K*, cock and discharge pipe for residue.

kettle *A*, which lies in the cast-iron pan *B*, and is heated to a certain temperature by a metal bath, generally of lead. The fatty acids, heated nearly to the boiling-point in a preparatory heater, are brought into the kettle *A*, through the funnel *C*, until the kettle is one-third full, the admittance of the fatty acid being regulated by the float-gauge *D*. Through the box *E*, which is provided with a safety-valve, the steam enters through the pipe *F*, which, on the lower end, branches off into two perforated arms.

The object of the steam is to drive the products of distillation into the condenser, the heating of the fatty acids being effected by the metal bath. The products of distillation escape through the helmet *G* into the collecting vessel *H* provided with a thermometer. The fat carried away with the steam collects in the collecting vessel, and can be drawn off through the cocks *J*, while the vapors loaded with fatty acids escape to the condenser. The condenser or refrigerator consists of a long copper box, with divisions extending alternately not entirely from the bottom to the lid. It is entirely submerged in cold water in a wooden vessel, the water being constantly renewed. The products of distillation enter on one end of the condenser, and leave it on the other; each division is provided with a discharge cock.

When the process of distillation is in full activity, which is readily recognized on the end of the condenser, new material is constantly introduced through the funnel *C*, the lead over the fireplace being constantly kept in a molten state and on the other end of the kettle in a pasty condition, which is the principal sign of the temperature being correct.

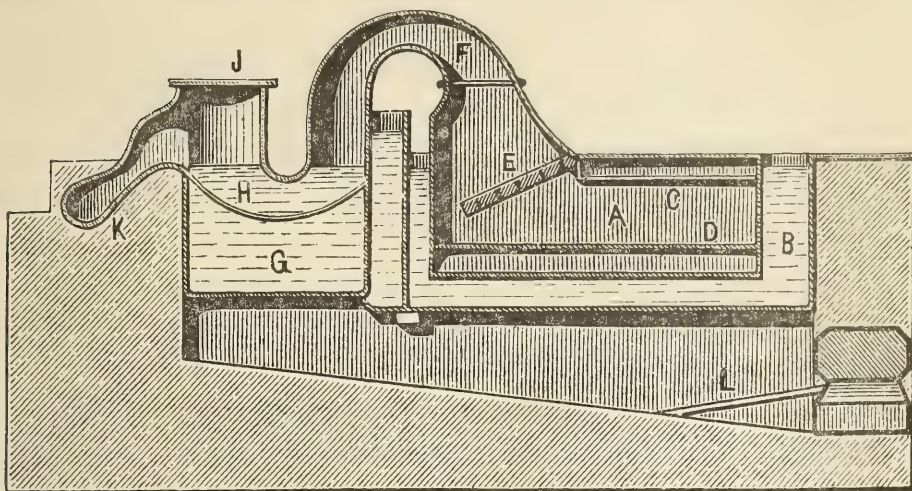
After distillation has been carried on for some time, the tarry residue, amounting to about one-tenth of the fatty substance, has accumulated to such an extent as to require its removal. For this purpose the admission of fatty acids is interrupted and distillation continued until no more volatile substances are developed. The valve at *H* is then closed and the cock at *K* opened, whereby the steam forces the tarry mass through the pipe at *K* into a suitable collecting vessel.

Schattenbach carries on the distillation in a cast-iron kettle, *A* (Fig. 11), divided into three divisions by the partitions *C*, *D*, and standing in a lead bath, *B*. The fat introduced into the upper division *C* falls in drops through a perforated bottom plate, while a current of superheated steam is forced against the jets of fats from the lower division *D* through a plate provided with cocks 0.38 inch in diameter. The carrying away of particles of fat is prevented by two other perforated plates, *E*. The vapors enter through the arched piece *F* through a small boiler, *H*, which is partly filled with hydrate of lime, and stands in the lead bath *G*, and is filled through the aperture *J*. The lime absorbing the



carbonic acid in the vapors, the latter are forced by the steam into the pipe *K*, which is connected on one end with a steam boiler

Fig. 11.



Schattenbach's Apparatus for Distilling Fatty Acids.

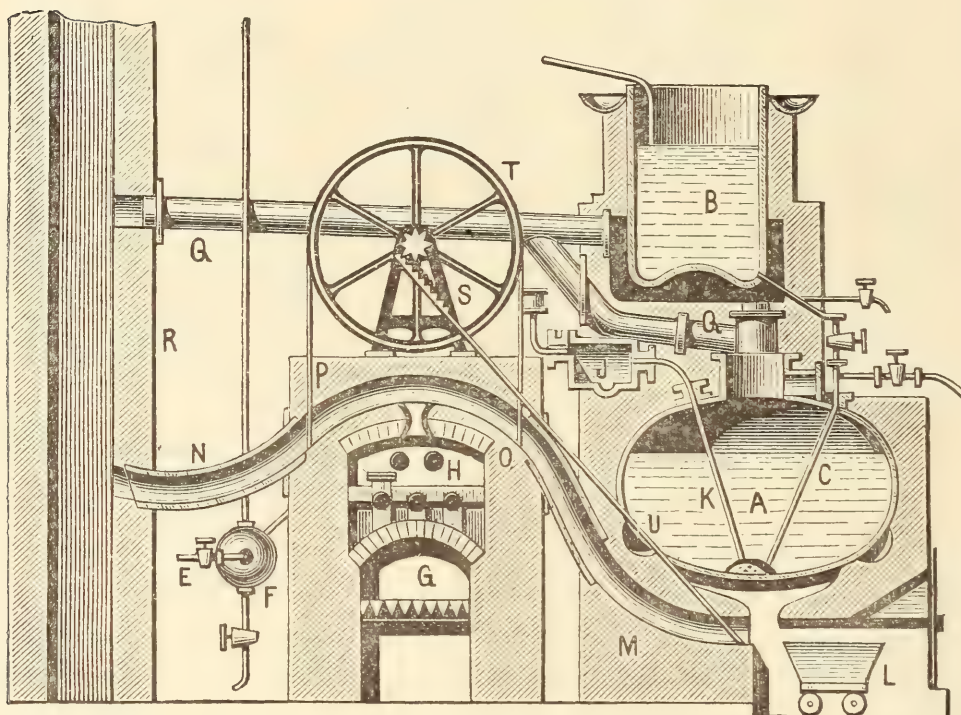
*A*, cast-iron kettle ; *B*, lead bath ; *C*, *D*, partitions ; *E*, perforated plate ; *F*, arched piece ; *G*, second lead bath ; *H*, small boiler ; *J*, aperture for filling *H* ; *K*, pipe ; *L*, fireplaces.

and on the other with a condensing apparatus. *L* are fireplaces for heating the lead baths.

Fig. 12 shows the vertical section and Fig. 13 the ground-plan of an apparatus for distilling fatty acids, first suggested by Tribouillet, of Neuilly, and which is much used notwithstanding its complicated construction. *E* is a steam pipe leading from the steam boiler, not shown in the illustration, to the steam reservoir *F*, from which the condensed vapors pass off through a pipe in the bottom. The vapors are heated by passing through the lateral pipe on the steam reservoir *F* into the pipes (superheater) *H* lying in a zigzag and horizontally over the fireplace *G*. They pass then through a pipe into the reservoir *J* for the subsidence of particles of fats which may have ascended from the still. On this reservoir is also a vertical pipe, on the upper end of which sits a valve opening inward and admitting external air in case a rarefied space should be formed in the reservoir *J* to the steam reservoir *F*, etc. For the introduction of the steam in as many

small jets as possible, a pipe *K* provided with a rose leads from the reservoir *J* to the still *A*. The latter is provided with a discharge pipe which carries the products of distillation into a coil cooled by water, and with the charging pipe *C* leading from the

Fig. 12.



Tribouillet's Distilling Apparatus for Fatty Acids. Vertical section.

*A*, still ; *B*, boiler ; *C*, conduit for fat ; *D*, discharge conduit ; *E*, steam conduit ; *F*, steam reservoir ; *G*, fireplace ; *H*, superheater ; *J*, reservoir ; *K*, pipe with rose ; *L*, wagon ; *M*, *N*, channels ; *O*, *P*, slides ; *Q*, funnel pipe ; *R*, chimney ; *S*, rack and pinion ; *T*, pulley ; *U*, metal rod.

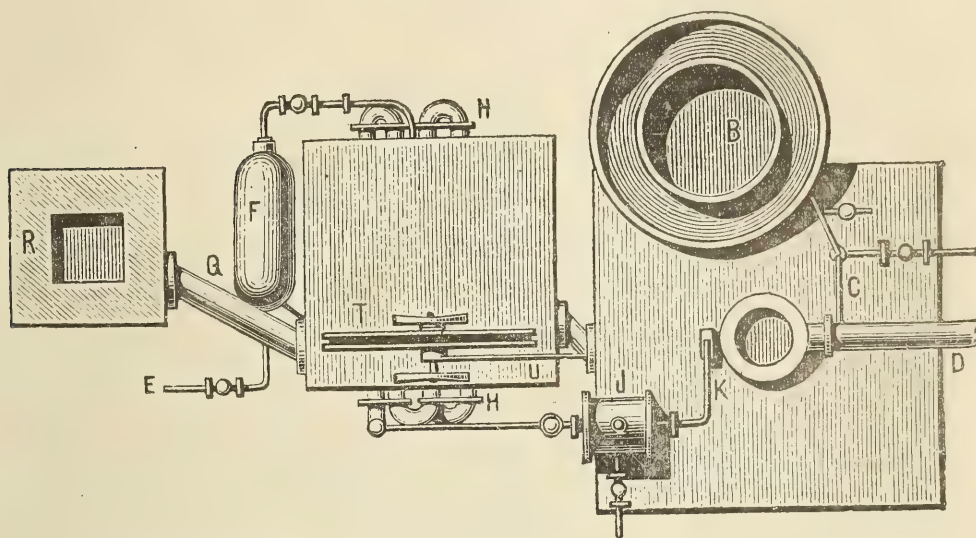
boiler *B*, which stands over the still and serves for the preparatory heating of the fatty acids. This boiler is provided all round with a gutter so that in filling it no fat can be spilled. By means of several cocks placed on the upper part of the pipe *C* the fat in the boiler *B* can be discharged outside, and the pipe *C* be used as a rising pipe for forcing upward and discharging through the lowest three cocks the fluid fat, or the brown, resinous, and tarry residues in the still, even when the discharge pipe remains closed



and the connection between the still *A* and the boiler *B* is interrupted.

The heating of the still *A* and the regulation of the heat is effected in the following manner: The hot gases, flames, etc.,

Fig. 13.



Tribouillet's Distilling Apparatus for Fatty Acids. Ground-plan.

from the fireplace *G*, pass through above the superheater *H*, where they have two escapes, to the right at *N* and to the left at *M*. In the beginning of the firing the channel *N* is closed and the hot current of air, passing through *M* beneath the still *A* and circulating in the flues, ascends behind the still underneath the boiler *B*, and, after heating the latter, passes into the chimney; or, *B* being sufficiently heated, it can be directly conducted into the chimney through the funnel-pipe *Q*.

The hot gases on their way from the superheater *H* to the still, meet on their exit from the channel *M* a metal rod *U*, which carries a toothed gearing connected with a rack and pinion, and causes the pulley *T*, sitting upon the same shaft, to revolve one way or the other, according as the rod *U* expands or contracts. Over the pulley *T* passes a chain to the end of which are secured the slides *O* and *P*. The rod becoming strongly heated expands and moves the pulley *T* so that the right hand slide is lowered, closing the channel *M* partly or entirely. In this case the hot gases pass directly through the channel *N* and the chimney *R*



until the rod *U* by cooling contracts sufficiently to lift the slide closing the channel *M*. To be enabled to heat the still *A* by a separate fire in the beginning of the operation, a door is fixed in the hearth in which the still is bricked, through which a small iron wagon *L* filled with glowing coals is pushed. The bottom of the wagon forms a grate, the whole being so arranged that the air entering from the outside has to pass through this grate. Before the introduction of the superheated vapors, the temperature of the still is brought to from  $482^{\circ}$  to  $572^{\circ}$  F., the correct temperature being recognized by a thermometer inserted in the still. The temperature of the vapors is also raised to the same degrees. The fatty acids run constantly from the boiler *B* to the still *A* until it becomes necessary to clean the still from the residues, which is effected by steam pressure.

Payen states that in distilling off a certain fatty substance, the products of distillation caught separately from time to time show different melting points, and, what is remarkable, that the melting temperatures of kitchen fat, bone fat, and beef tallow rise towards the end of the operation, while those of palm-oil sink the lower the longer distillation is continued.

The melting temperatures of the former rise from  $104^{\circ}$  to  $113^{\circ}$  F., while those of the latter sink from  $130^{\circ}$  to  $103^{\circ}$  F., as follows:—

	1.	2.	3.	4.	5.	6.	7.
Kitchen and bone fat	$104^{\circ}$	$105.8^{\circ}$	$105.8^{\circ}$	$107.6^{\circ}$	$113^{\circ}$	$113^{\circ}$	$116.6^{\circ}$
Palm oil . . .	130	125.6	118.4	114.8	111.2	105.8	103

The first products of distillation of palm-oil saponified with sulphuric acid are, therefore, so solid that no fluid acid can be expressed, and can be at once used for the manufacture of candles while the later products are only converted into cakes by remelting, pressing in hydraulic presses, and remelting the fatty acids with water. The fluid products running off from the press are used in the fabrication of soap. It is, however, necessary to remark that there is an essential difference between oleic acid obtained by distillation and that from saponification by lime; the

former, being contaminated by products of distillation, acrolein, etc., cannot be converted into elaïdic acid by nitrous acid, showing in that respect the same behavior as oxidized oleic acid.

The yield of products of distillation from fats is : From—

Suint . . . . .	47 to 55 per cent.
Olive-oil residues . . . . .	47 to 50 “
Palm-oil . . . . .	75 to 80 “
Fat from slaughter houses . . . . .	60 to 66 “
Oleic acid from stearin factories . . . . .	25 to 30 “

The brown residue resembling asphalt when cold amounts to 6 to 7 per cent. of the poorer qualities of fat, but only to 5 per cent. of palm-oil ; it can be used in the manufacture of gas and for the fabrication of poorer qualities of soap.

Finally, it may be mentioned that the products of distillation contain sebacic acid formed from the oleic acid.

3. *Decomposition of the fats by water.* a. *By high pressure and increased temperature.*—The idea of decomposing fats by superheated water is not a new one, for some hints to that effect were given by Appert (1825) and by Manicler (1826), though the aim of these technologists was a different one from the one at present demanded by the industry. Technically, the process was first executed by Tilghman, who, in 1854, took out a patent, while shortly afterwards Melsens, of Brussels, patented a similar process with the use, however, of sulphuric acid. Tilghman adds to the neutral fat to be decomposed one-third to one-half its volume of water, and pours this mixture into a sufficiently strong vessel, in which the fluids can be submitted to the action of heat, viz., a degree nearly as high as the melting point of lead, 588° F. The vessel is so arranged that during the operation it can be closed to prevent the evaporation of water. The original apparatus seems only to have been intended for experimental operations.

Payen and Wilson constructed an apparatus for the execution of the operation, and obtained the fatty acids and the glycerin in separate layers. After finishing the operation, and sometimes during the same, vapors surcharged with volatile fatty acids and acrolein frequently escape through a valve, and there is danger of an explosion, though no satisfactory reason can be given for it. The shattering of the apparatus generally takes place when the

temperature, and consequently the tension, in the apparatus is too high, and this evil is to be counteracted by opening the valve.

The most recent and ingenious apparatus constructed for this purpose is that of Wright and Fouché. It consists of two strong copper boilers, hermetically closed, and placed one above the other. They are connected by two pipes, one (*a*) of which reaches nearly to the bottom of the lower boiler, and ends in the upper one just above the bottom. The second pipe (*b*) is fixed in the lid of the lower boiler, and passes through the upper vessel, reaching nearly to its cover. The upper boiler is the steam generator, while the decomposition of the fats takes place in the lower one. For carrying out the operation, the steam generator is filled with water to the point where the first pipe (*a*) ends in it, while the second boiler is filled with the melted fat so that the latter reaches the top of the second pipe (*b*). There remains thus a free space, called by the patentee "*chambre d'expansion*" (expansion space). Heat being applied to the generator, the steam formed is carried by the second pipe (*b*) into the expansion space, and, becoming condensed, forces its way downward through the specifically lighter fat, and flows through the first pipe (*a*) again into the generator. In this manner the neutral fat substance is intimately and continuously mixed at a high temperature and at a high pressure with water, and completely disassociated in a short time into fatty acid and glycerin.

Melsens uses a Papin's digester, in which the fat to be decomposed is heated to from  $346^{\circ}$  to  $392^{\circ}$  F. with 10 to 20 per cent. of water, to which 1 to 10 per cent. of sulphuric acid has been added.

*b. Decomposition (saponification) of the fats by means of super-heated steam.*—This process is carried on on a large scale by the well-known Price's Candle Co., at Battersea, England, the suitable distilling apparatus having been invented by Wilson and Gwynne. By closely watching the temperature the fatty acids and glycerin are distilled over without undergoing any decomposition.

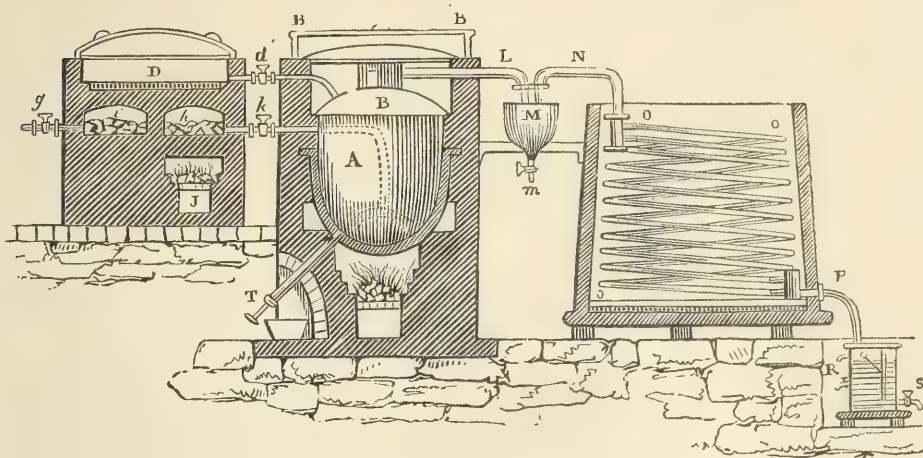
The apparatus used for distilling being nearly the same as those already mentioned, we will only describe Wilson's apparatus (Fig. 14).

The flat metal boiler *D*, with dome-shaped cover, is heated by



the waste heat of the superheater; the fluid fat is let into the copper boiler *A*, the cock *d* serving to regulate the flow. The

Fig. 14.



Wilson's Distilling Apparatus.

dome-cover *B* of the boiler has a cap of iron *BB*, which is heated with coals to prevent loss of heat by radiation. The steam being heated to from  $480^{\circ}$  to  $580^{\circ}$  F. and let into the boiler *A*, changes the fats into fatty acids and glycerin, and the vapors rising with the steam are carried into the pipes *L* and *N* and to the condenser *OO*. The steam excludes the air from the interior of the boiler, and thus promotes the decomposition.

According to the material a greater or smaller portion of the fatty acids obtained can be directly used for the fabrication of candles; the glycerin is purified by redistillation with steam. As previously mentioned, the temperature must be carefully kept at from  $480^{\circ}$  to  $580^{\circ}$  F., as by exceeding the latter degree the fat is decomposed, and an enormous development of acrolein takes place.

The fatty acids prepared by saponification with water and superheated steam excel in hardness and brittleness, and the candles made from them are transparent, and acquire a fine lustre by rubbing. The oleic acid obtained by this method is well adapted for the fabrication of soda and potash soaps.

*Glycerin*.—This is a by-product, in many of the processes above described, for the saponification and preparation of the fatty acids. It is obtained in the stearin factories as an aqueous

solution, showing at the utmost  $4^{\circ}$  to  $5^{\circ}$  B., and is more or less contaminated according to the manner in which it is obtained, sulphuric acid being the principal contamination from the acid saponification. To fix the latter add slaked lime until alkaline reaction takes place, and then evaporate the glycerin with indirect steam. During evaporation the greater portion of the calcium sulphate separates out, while a strong scum, containing many impurities, forms on the surface, and is removed with perforated ladles. The glycerin being evaporated to the desired concentration, generally  $25^{\circ}$  to  $28^{\circ}$  B., the steam is shut off, and, after allowing the calcium sulphate to settle, the supernatant clear glycerin is drawn off by means of a siphon. The sediment is washed with water and the latter evaporated with a new lot.

The evaporation of glycerin in open vessels, however, involving loss, some manufacturers evaporate in a vacuum, an apparatus with revolving cylinder, constructed by Leon Droux, being very suitable for the purpose. It will be described later on in speaking of the utilization of sub-lyes from curd soaps.

The glycerin-water obtained by the ordinary process of saponification by lime, or in the autoclave, is turbid from finely divided particles of lime, which cannot be removed by filtering. Hence the glycerin-lye is diluted with dilute sulphuric acid until it shows a slightly acid reaction; the lime-soap being decomposed and the fatty acids separating on top. The glycerin solution is then filtered, neutralized with calcium carbonate, and finally evaporated to the desired concentration.

The crude glycerin of commerce is of a more or less dark-brown color, and, to a greater or smaller extent, contaminated by volatile fatty acids, hydrocarbons, and inorganic salts, the latter originating from the lime and water used in the saponification. For many technical purposes crude glycerin can be at once used; for most, however, it has to be purified. This is done either by simple filtration over animal charcoal or by distillation with superheated steam, the product obtained by the first method being known in commerce as "refined" glycerin; it is, however, not entirely pure, as it always contains a larger or smaller quantity of lime salts and traces of butyric acid, the latter being recognized by the odor on rubbing a sample in the hand. Chemically pure glycerin can

be obtained only by distillation or by crystallization, the latter process having been recommended by Kraut and first technically utilized by F. A. Sarg & Son, of Liesing, near Vienna. The process is, however, too slow.

Chemically pure glycerin should not become turbid by the addition of ammonia and calcium oxalate, nor of nitric acid and nitrate of silver; it must not smell when rubbed upon the hand, nor become colored by the addition of nitrate of silver. The "so-called simple distilled glycerin" generally shows a slight turbidity on adding ammonia and calcium oxalate, as well as nitric acid and nitrate of silver. These slight traces of foreign salts do not injure the value of the glycerin for technical and other purposes.

Glycerin is now largely employed in many industries, such as for keeping clay moist for modelling, preventing mustard from drying up, keeping snuff damp, preserving fruit, sweetening beer and liquors; also for lubricating fine machinery; in fact it would take a large space to enumerate its many uses.

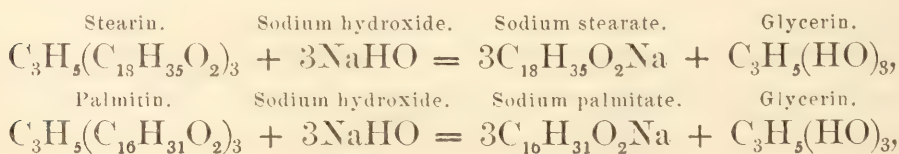
In the manufacture of soap the glycerin passes either into the soap or into the sub-lye. From the latter its recovery is difficult on account of the great content of salt; the methods used for the purpose will be given later on.

*Soaps.*—The ordinary soaps of commerce are, generally speaking, mixtures of the potassium and sodium salts of stearic, palmitic, and oleic acids, and if cocoa-nut and palm-oils are used as an addition, also of lauric acid. They are soluble in ether, benzene, and petroleum ether. Their alcoholic solutions are transparent and admit of filtering; at a certain degree of concentration they form a jelly. By evaporating them to dryness the soap is left behind as a clear, transparent, solid mass, free from crystalline admixtures.

Soaps show a peculiar behavior towards water. While they dissolve to a clear solution in boiling water, a solution in cold water is peculiarly turbid and opalescent. If, according to Knapp, a piece of soap is placed in a wire net or cloth, and suspended just below the level of cold water in a tall vessel, a certain amount of the constituents of the soap passes gradually into solution, while the residue remains as a coherent piece of the original



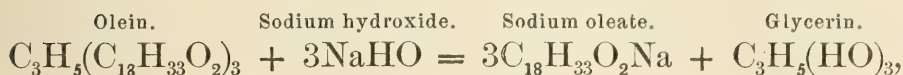
shape. This insoluble residue presents a woody, fibrous appearance, and forms, so to say, the skeleton of the original soap mass. By simply pouring cold water over a piece of soap and subsequent frequent shaking or stirring, it dissolves to a white, milky, opaque mass in which floats a turbid residue which, when moved in the light, frequently shows a remarkable silky lustre. The solution in cold water is, therefore, incomplete. There is some diversity of opinion about the processes which take place on treating soap with cold water. Chevreul found that by dissolving 1 part of potassium stearate in 5000 parts of cold water, potassium bistearate is separated in nacreous crystals and potassium hydrate remains dissolved in the water, while by using less water a mixture of neutral potassium and potassium bistearate is separated, and one-quarter of the quantity of the potassium remains in solution. These observations of Chevreul were applied to ordinary soaps, and it was held that soaps are decomposed by much water into acid salts, which separate, and into free alkalies, which remain in solution. This view is, however, contested by many chemists, especially by Rotondi,\* A. Fricke,† and M. Dechan and T. Maben.‡ Rotondi is of the opinion that soaps are not decomposed into alkaline bisabate and free alkali, but into alkaline sebate and a basic alkaline sebate. Fricke considers it probable that in tallow-curd soap the sodium oleate dissolves, while the sodium palmitate or stearate separates in nacreous threads. He further found that the residue left by lixiviating soap in cold water is even insoluble in hot water, and that alcohol dissolves only 0.48 per cent., it being, however, freely soluble in the hot solution of the extracted portion. Dechan and Maben are of the opinion that by the saponification of fats not only neutral salts are formed, so that the process is not only according to the following equations,



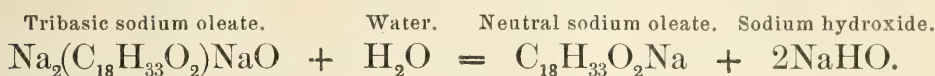
\* Seifenfabrikant, 1886, S. 284.

† Dingler. Polytech. Jour. 209, S. 46.

‡ Seifenfabrikant, 1886, S. 91.



but also basic salts, therefore, salts of the formula  $\text{Na}(\text{C}_{18}\text{H}_{33}\text{O}_2)\text{NaO}$ . By treating such salt with cold water, they claim, that decomposition takes place according to the following equation:—



Of the above opinions, we consider Rotondi's as the most correct. His experiments to determine the behavior of soaps towards water are very interesting and of importance as regards the question upon what depends the action of soap in washing.

He prepared a pure soap by precipitating a solution of good Marseilles (castile) soap with common salt, dissolving the precipitate in 95 per cent. alcohol, filtering, evaporating the alcohol from the filtrate, and repeating the operation. He obtained in this manner a soap completely soluble in boiling water, and showing an alkaline reaction with litmus, but containing neither alkaline hydroxide nor alkaline carbonate. It had the following composition:—

	Air dried.	Dried over sulphuric acid.
Water . . . . .	29.41	—
Fatty acids with a melting-point of 107° F.	64.90	91.43
Sodium oxide . . . . .	7.82	11.32
Inorganic salts . . . . .	0.16	0.31

From this soap Rotondi prepared solutions of various concentrations and subjected them to dialysis in the following manner: 1. 100 grammes of soap comminuted as much as possible were placed in 1 litre of distilled water and subjected to dialysis for thirty days at a temperature of 59° F. 2. 100 grammes of soap were brought into 2 litres of water and treated like No. 1. 3. 100 grammes of soap, dissolved in 2 litres of boiling water, were subjected to dialysis for thirty days at 59° F. 4. 100 grammes of soap in  $\frac{1}{2}$  litre of distilled water were subjected to dialysis, the temperature being kept every day at 176° F. After the expiration of the time the different diffused and non-diffused

fluids were evaporated in a water-bath ; the residues, after drying over sulphuric acid, gave the following results :—

Experiments.	Per cent. of sodium oxide in the residues	
	Of the diffused fluids.	Of the non-diffused fluids.
1 . . . . .	15.06	10.81
2 . . . . .	15.34	10.74
3 . . . . .	16.12	9.56
4 . . . . .	16.58	9.33

The quantity of diffused soap was very small in proportion to the non-diffused, especially in the first and second experiments.

The residues remaining after the evaporation of the diffused fluids are soluble in hot water, the solutions not becoming turbid except when exposed to the air for a few days. The residues of the non-diffused fluids are only soluble in hot water, and become turbid immediately after cooling. By continuing dialysis for a sufficient time, a residue is finally obtained which cannot be diffused and is insoluble in hot water. Since the diffused fluids contain no free alkali, Rotondi draws the following conclusions from the experiments :—

1. Neutral soaps with an alkali as a basis ( $C_nH_{2n-1}MO_2$ ) are decomposed by water into basic soaps ( $C_nH_{2n-1}MO_2OMH$ ), soluble in hot and cold water, and in acid soaps ( $C_nH_{2n-1}MO_2C_nH_{2n}O_2$ ) insoluble in cold and very sparingly soluble in hot water. 2. The decomposition of neutral soaps takes place more freely in hot than in cold water, and more rapidly or more slowly according to the concentration of the solution and the prevailing temperature. 3. The basic soaps diffuse freely, but the acid ones with difficulty. 4. The solution of basic soap obtained by dialysis may contain some neutral soap, which can be decomposed into basic and acid soap, until the solution finally contains only basic soap. 5. By the decomposition of neutral soaps by water neither alkaline hydroxide nor alkaline carbonate is liberated, which is readily shown by precipitating the neutral soaps with common salt and analyzing the fluid after filtering.

The action of water upon soap can also be established without dialysis. Rotondi prepared a solution of 100 grammes of soap in 1 litre of water by heating, and allowed it to stand in an open



vessel for twenty-four hours. By filtering, the solution could then be separated into an insoluble soap (*a*) and into a fluid one (*b*). The soap *a*, dried over sulphuric acid, gave a residue which contained 10.17 per cent. of sodium oxide. This residue was completely soluble in boiling water. By allowing, however, the solution to cool, and filtering, an insoluble soap remained behind, which contained 6.11 per cent. of sodium oxide.

The solution *b*, on evaporating, left behind a residue containing 14.12 per cent. of sodium oxide. By allowing it to stand in an open vessel, a fibrous substance secreted which in fourteen days was separated by filtration, and, after drying over sulphuric acid, contained 7.84 per cent. of sodium oxide. The evaporated filtrate yielded a residue which contained 15.97 per cent. By allowing it to stand fourteen days longer in an open vessel, a fresh quantity of insoluble substance formed; nothing more, however, separated from the fluid filtered off. On evaporation a residue was left behind which contained 16.21 per cent. of sodium oxide. It was completely soluble in cold water, showed a strongly alkaline reaction, could be completely precipitated with common salt, and the fluid filtered off from the precipitate contained neither alkaline hydroxide nor alkaline carbonate.

These experiments show that neutral soaps are decomposed by water into basic and acid soaps; that the former are completely soluble in cold water, while the latter are very sparingly soluble even in hot water; that the basic soaps are completely precipitated by common salt, whereby no alkali is liberated. The aqueous solution of basic soaps, especially when hot, yields, with oleic and other fatty acids, a clear fluid, which remains so on cooling, provided a sufficient quantity of basic soap be present. If the solution, however, remains in contact with air, it becomes turbid in a few days by the formation of acid and basic soap from the mixture of basic and neutral soap previously formed.

When heated the basic soaps dissolve the acid ones. The solution, on cooling, becomes turbid sooner or later, according to the proportion of basic and acid soap present. This explains why ordinary soaps are soluble in hot water, and decomposition is more complete than in cold water. Cold aqueous solutions of basic soaps do not combine with the fats, but only form emul-

sions with them. With triolein a white mass is obtained in this manner which looks like beaten cream and keeps for several months without the particles of oil separating. Heating produces no alteration in the mass. By adding, however, 80 per cent. alcohol, the fat separates on top as a clear fluid, which contains no glycerin; proof that the fat was not saponified, but formed an emulsion.

This property is also possessed, though in a less degree, by ordinary neutral soaps, especially in a hot solution, since they are then more readily decomposed into basic and acid soaps than in a cold solution. Saponification does not take place, as the fat can be readily separated by alcohol, and no glycerin is found in the alcoholic solution. The acid soaps do not possess the property of forming emulsions, even in the heat, or, at the utmost, in a very slight degree.

By conducting carbonic acid through a cold aqueous solution of basic soap, a milky fluid is formed, which, however, becomes again clear by standing in the air, heating, or the addition of alcohol. By filtration it is difficult to obtain a clear fluid. Common salt precipitates all the soap dissolved or suspended in the fluid, the residual fluid being free from alkaline hydroxide and alkaline carbonate. The insolubility of basic soaps in water containing carbonic acid explains why basic and neutral soaps (independent of the inorganic salts contained in them) possess less cleansing power when used with water strongly impregnated with carbonic acid.

When by evaporation water is withdrawn from soap solutions, they become at first thickly fluid and finally so viscid as to permit of the drawing of threads. On cooling the thickly-fluid solutions form, according to circumstances, either a jelly or a solid mass, both, however, containing a large quantity of water, a considerable portion of which is mechanically fixed, and another chemically, and is retained at  $212^{\circ}$  F. This affinity to water is very unequal in soaps of various fats and constitutes differences of great practical importance. It manifests itself, on the one hand, in the quantity of water the soaps are capable of fixing on congealing (as a rule soaps from solid fats can fix more water), and, on the other hand, in the behavior towards the mois-

ture of the air. Dry potash soaps strongly absorb water from the air, while moist soda soaps dry in the air. The nature of the fatty acids exerts a similar influence, soaps from oleic acid being far more hygroscopic than those from solid fatty acids. Exposed for some time to the air—

100 parts by weight of sodium stearate	absorb	7½ parts by weight.
100      “      “      potassium stearate	“	20      “      “
100      “      “      potassium oleate	“	162      “      “

Soaps from oleic acid, with potash as a basis, even if previously solid, swell up gradually to a jelly on exposure to the air.

The solid, hard mass congealed from soap solutions does not present a homogeneous body, provided its percentage of water does not exceed a certain degree; on the contrary, crystalline veins, formed, no doubt, by alkaline bistearate and alkaline bipalmitate, appear upon an opaque, amorphous ground. Soaps from cocoanut-oil, palm-kernel oil, and castor-oil form, however, an exception, since they congeal to an entirely amorphous mass.

In the presence of other bodies which have great affinity towards water and are readily soluble in it, the behavior of soaps towards water undergoes material alterations. A piece of soda soap floats in a cold concentrated solution of common salt like mercury. By heating the salt solution the soap softens to a thick, viscid mass, which floats on top, sharply separated from the salt solution, and on shaking separates into flakes, collecting, however, again when at rest. The soap yields thereby water to the salt solution, but only to a certain amount which it retains fixed. Soda soap is, therefore, insoluble in cold and hot concentrated solutions of common salt.

When solutions of soap and of common salt are poured together they mix only to a certain degree of dilution, which for most soaps must be very great, a more limited degree being allowable only for soaps from cocoanut-oil and palm-kernel oil. More concentrated solutions deposit themselves in separate layers over each other. By adding to an aqueous solution of soap common salt, the latter, for its own solution, withdraws a certain quantity of water from the former, separate solutions of salt and soap being thus formed.



Solutions of potassium acetate, ammonium chloride, calcium chloride, and sodium carbonate, and sulphate produce a similar effect, though in a less degree. In concentrated caustic lye most soaps are insoluble ; all are, however, soluble in weak caustic lye.

Potash soaps are decomposed by sodium salts, for instance, common salt, Glauber's salt, etc., the corresponding potassium salt (potassium chloride, potassium sulphate) and soda soap being formed. For a long time this was the only method of preparing hard soaps in Germany ; the fat was saponified with potash lye and common salt added to effect an exchange of bases, which, however, was never complete, the soap thus prepared containing always potash, and being in consequence somewhat softer and more soluble than that made with pure soda lye. This process is still in vogue in countries where wood is burnt as fuel and potash and wood ashes are abundant : notably in Russia and the newly-settled portions of the United States. Germany now employs the artificial soda in almost all its soaps, and is making much of its alkali for the use of the soap-boiler as well as of the glass manufacturer from cryolite.

It was formerly attempted to explain the detergent properties of soap on the basis of Chevreul's experiments, mentioned on page 68. The alkaline salts of the fatty acids, it was claimed, were decomposed by cold water into alkaline bisbate insoluble in water, and into free alkali soluble in water. The liberated alkali, it was said, then exerts a dissolving influence upon grease and dirt, and, being sufficiently diluted, without attacking the skin or clothes, while at the same time the bisbates separated by the water, but suspended in it, envelop the grease and dirt dissolved from the clothes and prevent them from being again precipitated upon the fibre. That this theory is incorrect will be seen from the fact, that the decomposition into alkaline bisbate and free alkali is only complete in the presence of much water. If, therefore, Chevreul's opinion were correct, washing could be better accomplished with much water and little soap than with much soap and little water, which, however, is not the case. Further, the above-mentioned decomposition takes place only in cold water, but not in hot, in which the soap is, at all events, not decomposed into acid salt and free alkali. Cold water and soap

would, therefore, be more effective in washing than hot water and soap, while the reverse is the case. We are, therefore, inclined to accept Rotondi's theory that the soaps are decomposed by water into basic soaps which are soluble in cold and still more freely in hot water, and into acid soaps, and that the basic soaps have the property of converting the fats into an emulsion.

Knapp's theory deserves attention. He is of the opinion that the detergent action of soap is principally due to the great power of wetting substances possessed by soap solutions, which in this respect surpass nearly all other liquids. The dissolving effect of soap upon grease and dirt presupposes an extremely intimate contact between the substance to be dissolved and the solvent, and this contact is rendered possible by the wetting power of soap solutions. Soap-water penetrates tissues, etc., with greater ease and more completely and wets the surfaces more thoroughly than mere water; it readily dislodges the condensed layer of air upon the surface, and by capillarity forces itself between the surface of the substances to be cleansed and the adhering particles of dirt, dissolving and removing them.

A very original explanation of the detergent powers of soap has been advanced by Professor Jevons, of London. He observed that inorganic as well as organic particles suspended in water remain in a peculiar shaking and trembling motion, provided their diameter does not exceed  $\frac{1}{100}$  inch. To this phenomenon he applied the term "pedesis," a Greek word meaning a motion by leaps or bounds. Upon this fact Jevons bases his explanation of the effect of soap. We would, therefore, have to suppose that the particles of soap suspended in the water dash at the particles of dirt, thus dissolving and washing them away. Nearly all substances soluble in water have, it is claimed, the power of interrupting pedesis, which, according to Jevons, explains the fact that distilled water or pure rain-water possesses great cleansing power, since it produces a high degree of pedesis, while the comparatively small effect of hard water is due to a considerable decrease of pedesis, in consequence of the earthy salts dissolved in the water. It is difficult to come to a decision about the value or non-value of Jevons's theory; for us it is too artificial.

## CHAPTER III.

OCCURRENCE AND MANNER OF OBTAINING AND PURIFYING  
FATS AND FAT OILS.

*Occurrence and manner of gaining fats and oils.*—The fats are widely distributed throughout the animal and vegetable kingdoms. In the animal organism fat occurs in all tissues and organs and in all fluids except normal urine. In plants it is found partly scattered throughout the entire plant and partly heaped up in certain organs, especially in the seeds and cotyledons.

The vegetable fats found in moderate climates are liquid at an ordinary temperature. They are obtained by comminuting the seeds and subjecting the mass to strong pressure, or extracting it with a solvent (carbon bisulphide, petroleum ether, etc.) which can be readily volatilized from the oil.

Most of the vegetable fats occurring in warm climates are solid at our ordinary temperatures. They are mostly obtained in the country of their origin by boiling the oleaginous seeds and fruits with water. Some of them, such as palm-kernels, copra (the dried pulp of the cocoanut), etc., are also exported to Europe and America, where the fat is obtained by pressure or extraction.

The fat from animal substances is generally obtained by heating over an open fire or with steam. When the latter is used its action is generally assisted by the addition of acids or caustic alkalis. From bones the fat is generally extracted with benzene.

*Purifying and refining fats and oils.*—The fats and oils, no matter by what method they are obtained, are never entirely pure; they always contain more or less foreign substances derived from the raw materials, which form an obstacle in their use for many purposes. The animal fats contain generally particles of flesh and blood, which, if not removed, would in a certain sense act as



ferments and accelerate rancidity. The fats are, therefore, purified by remelting upon water over an open fire or with the assistance of steam.

Vegetable oils contain more or less mucus and other substances in solution, which give to the oil a milky turbidity. These foreign substances exert a disturbing influence if the oil is to be used for lubricating or illuminating purposes, it becoming soon rancid, and in burning chokes up the wick and deposits soot. It is, therefore, subjected to a purifying process called "refining," and the oil thus treated is termed "refined oil."

Oils for illuminating purposes are best refined by means of concentrated sulphuric acid, the quantity required varying according to the condition of the oil, 1 to  $1\frac{1}{2}$  per cent. being generally used when the oil is treated at an ordinary temperature. The oil must, however, not be too cold, as it then becomes too thickly fluid, and the substances carbonized by the sulphuric acid deposit themselves with difficulty.

The sulphuric acid is added in a thin stream and with constant stirring. The oil assumes a dark green color. Stirring is continued until a drop of oil placed upon a porcelain plate shows that the carbonized substances have coagulated to a black flake floating in the clear yellow oil. Stirring is then discontinued and the oil allowed to stand quietly six to twelve hours. When the carbonized substances have deposited themselves on the bottom the oil is drawn off into a large vat, and one-fourth to one-third of its volume of hot water added. It is then stirred moderately for a quarter of an hour, and allowed to stand until the oil separates from the water. The latter is then drawn off through a cock placed immediately above the bottom of the vat, and the washing of the oil repeated once or twice to remove the last traces of sulphuric acid adhering to it. For the complete attainment of this object, it is recommended to add some lime-paste or chalk to the water for the neutralization of the sulphuric acid. Such addition is, however, not necessary, and is rather disadvantageous since the lime is apt to saponify some of the oil, and chalk forms a disagreeable froth in consequence of the development of carbonic acid. A small quantity of soda may, however, be added to the last wash-water.

After the separation and drawing off of the oil from the last wash-water, it still contains some aqueous particles in suspension. They are deposited, and the oil rendered entirely clear by storing for some time at not too low a temperature. Clarification is effected still more quickly by adding some common salt to the oil before storing it.

Treatment with sulphuric acid is, no doubt, the best method for refining oils for illuminating purposes; for oils for lubricating purposes it is, however, not suitable, or at least oils thus refined must be subjected to special treatment. While the opinion, so frequently expressed, that oils treated with sulphuric acid always retain some of it, is not correct, because the sulphuric acid can be readily removed by boiling with water, oils thus refined always contain free fatty acids, and must, therefore, be rejected as lubricants, if not previously subjected to a special process to free them from acid. This is effected by various methods. One frequently employed consists in treating the oil with calcium carbonate (marble powder). This method is, however, not suitable, as the carbonate of lime will only remove traces of sulphuric acid, but not the free fatty acids. By another process the oil is treated with a few per cent. of lye. This, however, has the disadvantage that the soap-paste formed deposits with difficulty. It is, therefore, best to use concentrated solution of soda and boil the whole with steam.

To avoid the formation of free fatty acids, Bareswil has proposed to treat the oils with alkalis instead of acids. The oil is compounded with 2 to 3 per cent. of concentrated potash or soda lye, then thoroughly stirred and gradually heated. If, however, pure caustic lye is used, we have again to contend with the evil that the oil clarifies with difficulty. It is therefore better to use lyes not entirely caustic, and even in this case clarification is frequently connected with difficulties, and entirely clear oil is generally only obtained by filtration, which is usually executed through layers of gravel, the upper one consisting of coarse gravel, the next of finer, and the last of sand.

*Bleaching of oils and fats.*—The methods used for bleaching oils and fats are generally the same as in other industries: air and light and chemical agents; among the latter being lye, sul-

phuric acid, sulphurous acid, chlorine, potassium permanganate and potassium bichromate in connection with acid.

Light has an oxidizing effect upon all organic coloring substances, this action depending partly on an oxidation and a dis-oxidation of the coloring matter. Of great importance is the action of sunlight upon volatile and fat oils, as they are not only bleached, but the first are also quickly resinified, and the latter soon become rancid. Small quantities of oil are frequently bleached by exposure to air. For this purpose the oil in glass balloons or in flat zinc boxes is exposed to the sunlight. On a large scale bleaching with air and light is frequently employed for palm-oil, to which we will refer later on.

In soap factories lye is much used for bleaching linseed and cottonseed-oil, 5 to 10 per cent. of potash lye of  $28^{\circ}$  to  $30^{\circ}$  B., according to the condition of the oil, being usually employed for the purpose. The oil is first heated, best by direct steam, and the lye then added with vigorous stirring. The soap-paste separated in allowing the mass to rest is best used for dark soft soaps. Where the demand for the latter is a sufficiently large one, bleaching with lye is simple and cheap. But where the demand is chiefly for pale, transparent soft soaps, bleaching with sulphuric acid is preferred. For 2000 pounds of oil 60 to 80 pounds of sulphuric acid of  $66^{\circ}$  B., previously diluted with 20 pounds of water, are used. (In diluting sulphuric acid with water, the acid is poured into the water.) The diluted acid is then poured in a thin stream into the oil, stirring constantly. The oil soon acquires a dark green color, and stirring is continued for twenty minutes. 120 to 160 pounds of warm water are then poured through a rose over the oil and thoroughly mixed with it by stirring. The oil now assumes a pale green color, which indicates the success of the bleaching process. The oil is then covered and allowed to stand for a few days for clarification. It has then a milky-white appearance, due to finely divided particles of water, and can be immediately used for the manufacture of pale soft soaps.

For bleaching with sulphurous acid it is best to use sodium bisulphide, a concentrated aqueous solution of which exerts a powerful bleaching effect, but still more so by compounding it with some sulphuric acid; the latter should be diluted and very grad-



ually added, as otherwise a too violent development of sulphurous acid is introduced. For bleaching 100 pounds of oil 1 to  $1\frac{1}{2}$  pounds of sodium bisulphide are required.

For bleaching with calcium chloride pour about ten times its weight of water over it, let the mixture stand for some time, frequently stirring it, and then allow the sediment to settle. Of the supernatant liquor add, with constant stirring, to the oil to be bleached (previously compounded with some sulphuric acid) until the oil appears to be sufficiently bleached. It is then washed with hot water and clarified by filtering. This method of bleaching cannot, however, be recommended on account of the chlorine developed attacking the oils and fats too strongly.

Potassium permanganate, in connection with sulphuric acid, was formerly much used for bleaching palm-oil. A solution of the permanganate strongly acidulated with sulphuric acid was added with constant stirring to the melted palm-oil, and the stirring continued  $\frac{1}{2}$  to 1 hour. The oil was then allowed to rest until the next day. The bleached oil deposited itself upon the brownish fluid; this was removed and the oil repeatedly washed with hot water and then allowed to settle: pyrolusite (peroxide of manganese) was frequently substituted for potassium permanganate. To the heated oil compounded with dilute sulphuric acid was added in small quantities and with constant stirring finely pulverized pyrolusite, until the mass, at first black, assumed a light appearance. The process was then finished in the same manner as with the potassium permanganate.

Both of these processes are now, however, generally superseded by that of bleaching with potassium bichromate, which was first published by Watt in 1836. While he used potassium bichromate and sulphuric acid, hydrochloric acid is now generally substituted for the latter. Watt recommended the process, not only for palm-oil, but also for tallow and other fats. Its execution is similar to that with potassium permanganate and sulphuric acid, and we will refer to it more fully under palm-oil. Watt also published a process to regain the potassium chromate used. The green lye formed beneath the fat is drawn off, diluted with water, and then compounded with thick lime-paste until the sulphuric acid is saturated, *i. e.*, until the acid reaction disappears.

The formed gypsum is then allowed to deposit, the clear green solution drawn off, and after washing it once more, carefully precipitated with milk of lime until the green color disappears. The precipitate is thoroughly washed, dried, and heated to a red heat upon an iron plate. The powder gradually becomes yellow, and can be used for fresh bleaching operations by compounding it with sulphuric acid so that the latter slightly preponderates. This process of regaining the bleaching agent will of course only pay where very large quantities are used.

Since peroxide of hydrogen is now manufactured on a large scale, it is recommended as a bleaching agent for oils. The aqueous solution brought into commerce contains ten volumes of the peroxide, and must be intimately mixed with the oil. It does not act very energetically and much slower than the bleaching agents previously mentioned. Upon many oils it exerts no bleaching effect whatever; linseed-oil, for instance, which was beautifully bleached by sulphuric acid, was scarcely touched by peroxide of hydrogen.

Animal charcoal has also been recommended for bleaching oils; it is, however, not well adapted for the purpose, as it very quickly loses its bleaching power.

## CHAPTER IV.

## EXAMINATION OF FATS AND FAT OILS.

IN examining fats and fat oils problems of varying natures may present themselves for solution. It may be required to determine the chemical constitution of a fat, its identity, its percentage of solid and liquid glycerides, glycerin, and free fatty acids, and an intentional or unintentional contamination with other fats or oils, or with foreign admixtures. The solution of the first problem—the chemical constitution of a fat or oil—belongs to science, and therefore does not require our attention here; the other examinations are, however, frequently required in the fat industry, and we will, therefore, refer to various methods in use. They may be divided into three classes: 1. *Organoleptic methods*; 2. *Physical methods*; and 3. *Chemical methods*.

1. *Organoleptic methods*.—The organoleptic means, *i. e.*, odor, taste, and color, are generally employed in commerce as criterions of the quality of an oil. Their use requires, of course, great experience, and, besides, they are by no means reliable, since the color, taste, and odor of oils not only undergo alteration by age, but vary according to derivation; linseed-oil from Russian seed having, for instance, a different taste from that from Indian seed. The odor of an oil is generally tested by rubbing a few drops upon the palm of the hand. It has also been proposed to carefully heat a few drops of the oil to be examined in a small porcelain dish, and when sufficiently cool to rub some upon the palm of the hand and smell, carefully comparing the odor evolved with that arising from a known pure sample of the same kind and quantity of the oil similarly treated.

*Physical methods*.—Of the physical properties it has been attempted to utilize the following in testing oils: Cohesion, viscosity, specific gravity, melting and congealing points, solubility in glacial acetic acid, and electric conductivity.



Under phenomena of cohesion we understand, in this case, the form a drop of oil assumes in falling upon a glass plate or the flat, smooth bottom of a porcelain dish. Chatin and Thomlinson, who made these forms the subject of special investigation, found that certain varieties of oil, and even mixtures, assume definite forms by their particles holding closer together or spreading out more on the edge.

The oils, as previously mentioned, are distinguished at an ordinary temperature by a certain degree of *viscosity*. This can be readily ascertained by noticing the time required by equal quantities of oil to flow at the same temperature from an aperture of known width.

The determination of the *specific gravity* is of greater importance for testing oils than the preceding methods. Special areometers, so-called oleometers or oil-balances, have been constructed for commercial use. They are so weighted or graduated as to adapt themselves to the densities of the leading fixed oils. The instruments in general use are Gobby's and Lefèbvre's oleometer, Fisher's oil-balance, and Brix's areometer for lighter liquids. The scale of Gobby's oleometer is divided into  $50^{\circ}$ , and it floats at  $0^{\circ}$  or zero in pure poppy-oil, at  $38^{\circ}$  or  $38.5^{\circ}$  in pure almond-oil, and at  $50^{\circ}$  in pure olive-oil. The standard temperature of the instruments made in England is now  $60^{\circ}$  F.; of those made on the Continent,  $54.5^{\circ}$  F. The oil must therefore be brought to this normal temperature before testing it by plunging the glass cylinder containing it into either hot or cold water, as the case may be; or a correction of the observed density must be made. The last is done by deducting two from the indication of the instrument for each degree of the thermometer above the normal temperature of the instrument, and adding two for every degree below it. Thus: Suppose the temperature of the oil at the time of the experiment is  $60^{\circ}$  F. and the oleometer indicates  $61^{\circ}$ ; then—

$60.0^{\circ}$  actual temperature.

$54.5^{\circ}$  normal temperature.

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$5.5^{\circ}$  difference.

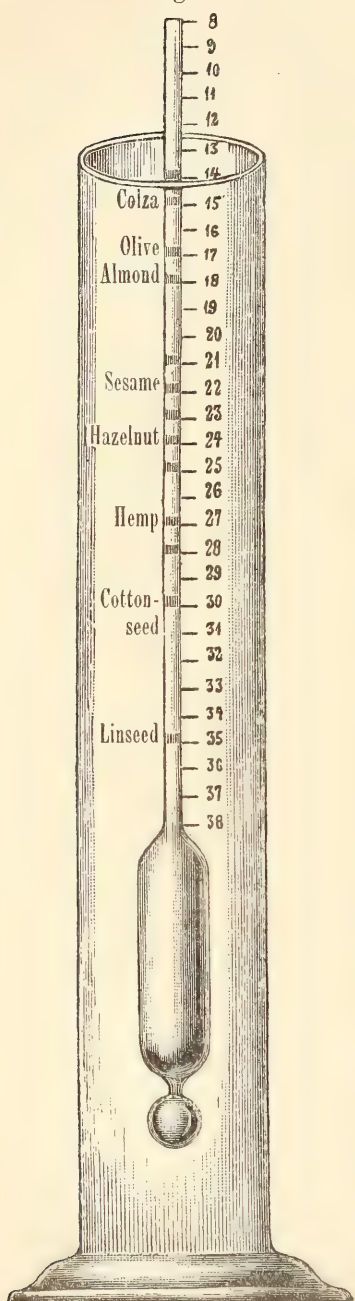
Indication of the oleometer  $61.0^{\circ}$

The difference  $5.5 \times 2 = 11.0^{\circ}$

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Real density,  $50.0^{\circ}$

Fig. 15.



Upon the scale of Lefèbvre's oleometer (Fig. 15) are indicated the specific gravities of the principal oils occurring in commerce. As the space is too limited to place four figures alongside each other, the first and last decimals are omitted. On the place for colza oil, for instance, stands the figure 15, which must be read 0.9150 specific gravity. On the left of the scale, opposite to the figures of the specific gravity, are the names of the various oils. To facilitate the reading, the oils are indicated by a color as near alike as that acquired by them by the test with sulphuric acid. The standard temperature of the oleometer is  $15^{\circ}$  C. ( $59^{\circ}$  F.). If oils are tested at a different temperature, the difference in the specific gravity is 0.001 more or less for each  $1.5^{\circ}$  C. above or below the standard temperature; therefore 0.002 for every  $3^{\circ}$  C., 0.004 for every  $6^{\circ}$  C., and so on. At  $18^{\circ}$  C. ( $64.4^{\circ}$  F.)  $\frac{2}{1000}$  must consequently be added to the specific gravity, and at  $12^{\circ}$  C. ( $53.6^{\circ}$  F.)  $\frac{2}{1000}$  subtracted.

Although, generally speaking, oil-balances give good results in testing oils as to their purity, the statements cannot be implicitly relied on. The specific gravities of oils vary very little, and exact experiments have shown that the variations of the specific gravity of one and the same variety of oil are frequently as great as the difference between the oil and another used as an adulterant. To this must further be added the difficulty of executing exact areometrical measures with such thickly-fluid substances as oil. To overcome this, Laurot, in 1841, constructed

an oil-balance for testing the oils at  $212^{\circ}$  F., but this proved of little service, especially since Scharlach has shown that oils do not expand equally by heating. The determination of the specific gravity as a means of testing fat oils is therefore limited to a few cases, and, generally speaking, cannot be considered reliable.

For the determination of the *melting-points* of fats various methods have been proposed, of which the following are the most important: 1. Suck the melted fats into capillary tubes, and after allowing the fat to congeal, place the tubes in a vessel with water. Heat slowly and note by a thermometer dipped into the water the temperature at which the fat becomes transparent; this is the melting-point. 2. The melted fat, according to Bouis, is brought into capillary tubes open on both ends. When the fat is congealed, the tubes are placed under water and heat is slowly applied. The melting-point is that degree at which the fat is pressed upwards in the tube. 3. According to Pohl, the bulb of a thermometer is coated with a layer of fat about three millimetres thick, and after allowing the fat to congeal the thermometer is placed in water. Heat is then slowly applied and the temperature noted at which the fat becomes transparent or melts off.

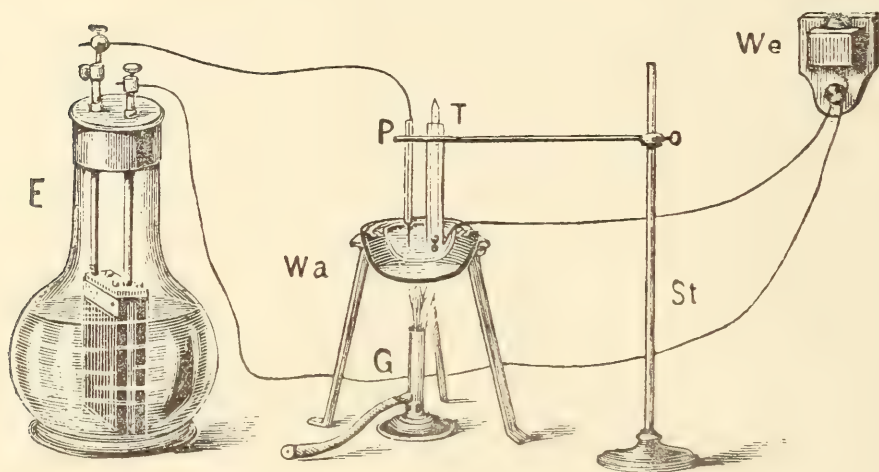
The last method gives, according to Ruedorff, the best results, though that none of them is entirely reliable is shown by the great variation in melting-points of fats as given by the various observers, the statements in regard to the melting-point of tallow varying, for instance, between  $98.5^{\circ}$  and  $139^{\circ}$  F.

A method first proposed by Julius Löwe gives always the same results. It is based upon the fact that an electric current of low power interrupted by a layer of fat can set in motion an alarm as soon as the fatty substance ceases to prevent the passage of the current. The apparatus (Fig. 16) used consists of a porcelain capsule about two inches deep and three and a half inches wide, filled with mercury, which is placed in a small copper water-bath, *Wa*. By means of a clamp attached to a retort-stand, *St*, an accurate thermometer, *T*, inclosed within a glass tube (to avoid changes of temperature by the surrounding air) is immersed in the mercury. By means of the same retort-stand a rather thick platinum wire, *P*, is firmly fixed so as to dip a short



distance into the mercury. One end of this wire is bent into an eye, which is connected with the copper wire of the zinc pole of the battery. The platinum wire itself is inserted into a glass tube, and secured with sealing wax for the purpose of keeping it straight and rigid. For the continuation of the current another thinner platinum wire dips into the mercury and connects with a copper wire leading to the alarm. The other pole of the bat-

Fig. 16.



Apparatus for determining Melting-Points.

tery is in direct connection with the latter. If the apparatus is properly constructed, the alarm should instantly sound on closing the current. Having made this preliminary trial, the stout platinum wire *P* is disconnected, its lower end, which had dipped into the mercury, strongly ignited and allowed to cool under mercury, after which it is quickly plunged into the almost melted fat substance the melting-point of which is to be determined.

The coating need not be thicker than a few millimetres in order to interrupt the current. On now re-connecting the platinum wire with the apparatus as before, the current cannot pass and the alarm will not sound. But if the water-bath, and with it the mercury, be gradually heated, a point will be reached at which the coating around the end of the wire melts, and by the re-establishment of the current the alarm will be sounded. At this moment, which indicates the actual melting-point of the substance, the temperature is read off.

For the determination of the *congealing point*, the fat is first melted at as low a temperature as possible, so that particles of unmelted fat still float in the melted portion; it is then allowed to congeal with constant shaking. By noting from minute to minute the temperature by means of a thermometer, it will be seen that with some fats the temperature sinks to a certain degree, and after remaining constant for some time sinks still further. As the fat congeals during the constant temperature, the latter must be considered the congealing point. With other fats the temperature sinks to a certain degree while they congeal more and more, but then rises several degrees, whereby the fats become entirely solid. With some of these fats the maximum to which the temperature rises is constant, and is, therefore, to be considered as the congealing point; other fats do not show this constancy, and an exact determination of their melting-points is, therefore, impossible.

E. Valenta has recommended the *behavior of fats and fat oils towards glacial acetic acid* as a means of testing them. By experiments he ascertained that most animal and vegetable fats are more or less soluble in glacial acetic acid, but that the behavior of the various oils varies so much that it can be advantageously utilized for their characterization. He made his experiments by intimately mixing in a test-tube equal parts of oil and glacial acetic acid, and exposing the mixture to various temperatures. Of the examined fats dissolved—

1. Completely at an ordinary temperature ( $59^{\circ}$  to  $68^{\circ}$  F.): olive-kernel oil and castor-oil.
2. Completely or nearly completely at from  $73.5^{\circ}$  F. to the boiling temperature of the glacial acetic acid: palm-oil, bay-oil, expressed nutmeg-oil, cocoanut-oil, palm-kernel oil, illoopa-oil or Mahwah butter, olive-oil, cocoa-oil or butter, sesame-oil, pumpkin-seed oil, almond-oil, cotton-seed oil, peanut-oil, Himalayan apricot-oil, beef-tallow, American bone-fat, cod-liver oil.
3. Incomplete at the boiling temperature of glacial acetic acid: colza-oil, rape-oil, hedge-radish oil.

For distinguishing the separate fats of the second group, equal volumes of fat and glacial acetic acid were slowly heated in a test-tube, with frequent shaking to a completely clear solution, then allowed to cool, and, by means of a thermometer dipped in the fluid, the temperature determined at which the clear solution commenced to become turbid. The following table shows the average results of these observations :—

Name of the fats.	Specific gravity at 59° F.	Solution in equal parts of glacial acetic acid becomes turbid at	Remarks.
Palm-oil . . . . .	—	73.5° F.	Fresh fat.
Bay-oil . . . . .	—	79–80.5	Old, rancid fat.
Expressed nutmeg-oil	—	80.5	— —
Cocoanut-oil . . . .	—	104	— —
Palm-kernel oil . . .	—	118.5	Old, rancid fat.
Illoopa-oil or Mahwah butter	0.9175	148	Obtained in the laboratory from the seeds of <i>Bassia longifolia</i> .
Green olive-oil . . .	0.9173	185	Oil from the second pressure; contains very likely a considerable quantity of olive-kernel oil.
Cocoa butter . . . .	—	221	
Sesame-oil . . . . .	0.9213	224.6	
Pumpkin-seed oil . .	0.9241	226.5	Crude Hungarian oil.
Almond-oil . . . . .	0.9186	230	From sweet almonds.
Cotton-seed oil . . .	0.9228	230	
Olive-oil (yellow) . .	0.9149	231.5	Oil from first pressure.
Peanut-oil . . . . .	0.9193	233.5	
Apricot-oil . . . . .	0.9191	237	
Beef tallow . . . . .	—	203	Beautiful, hard tallow from Italian trade.
American bone fat . .	—	194–203	
Cod-liver oil . . . .	—	213	

Notwithstanding the fact that the temperatures at which the turbidity of the solution of glacial acetic acid commences are subject to considerable variations, chiefly due to the varying percentage of free fatty acids in the fat, this method is very useful for testing oils, especially in connection with others.

Rousseau many years ago made attempts to utilize the *electrical conductivity* of oils for testing them. His method was based upon the fact that all fat oils, with the exception of olive-oil, are good conductors of electricity. He constructed a special appa-



ratus, which he termed "*diagometer*;" but the results obtained were not sufficiently accurate. Rousseau's idea has been recently again taken up by Palmieri, but scarcely with any better results.

*Chemical methods.*—The chemical methods employed for the examination of fats are based, 1, upon the fact that oleic acid is converted by nitrous acid into solid elaidic acid, while linoleic acid is not altered; 2, upon the increase of temperature the oils show with concentrated sulphuric acid; 3, upon the fact that the different oils can fix a varying quantity of calcium hydrate; and 4, that they can fix a varying quantity of iodine. Other chemical reactions used refer more to immaterial constituents which the fats have absorbed or retained in the evolution from the vegetable or animal body from which they are derived.

The simplest manner of executing the *elaidin test* is to place a few drops of the oil to be examined upon water in a test-tube, and introduce gas developed by the action of nitric acid upon iron filings. The oleic acid of non-drying oils is converted into a rigid mass, while the drying oils separate, according to their quantity, in drops or as liquid layers upon the surface. The color of the elaidin formed is characteristic for various oils, though it cannot be implicitly relied upon, since there is a possibility of the drying oils not being distinctly separated on account of being enveloped by the drying ones.

*The increase of temperature arising from the admixture of concentrated sulphuric acid* with the fatty oils as a test of their purity was first proposed by M. Maumené. Fehling, Casselmann, and Allen re-examined the subject, and in a treatise recently published by Maumené, he considers this method as the only one giving reliable results. While we cannot agree to this opinion, and do not consider the method sufficient for all cases, it cannot be denied that it gives good results, to distinguish, for instance, drying from non-drying oils, as the former become far more heated than the latter.

The test is executed as follows: 50 grammes of the oil are taken, the temperature is noted, and 10 cubic centimetres of sulphuric acid of 1.845 specific gravity are gradually added with constant stirring with a thermometer until the temperature ceases to rise. The following are the results obtained by Maumené.

	Rise of temperature.		Rise of temperature.
Olive-oil . . .	108° F.	Gingelly-oil . . .	154° F.
Castor-oil . . .	116	Poppy-oil . . .	187.5
Neat's-foot oil . . .	122.7	Hemp-oil . . .	208
Bitter-almond oil . . .	125	Nut-oil . . .	214
Sweet-almond oil . . .	126.5	Ray-liver oil . . .	215.5
Rape-oil . . .	134	Cod-liver oil . . .	215.5
Beech-nut oil . . .	149	Linseed-oil . . .	217
Peanut-oil . . .	152		

In the application of this test constancy is only to be looked for when the process is in each case performed strictly in the same way, and a test for comparison is, at the same time, made under the same conditions with a typical oil.

Of great importance for the examination of fats is the *determination of the saponification equivalents*. This method was first used by Koettstorfer to determine the adulterations of butter and other fats. He saponified 1 to 2 grammes of filtered butter fat in a tall, covered beaker glass of about 70 cubic centimetres capacity with 25 cubic centimetres of alcoholic potash lye of about one-half normal strength by heating in a water-bath for fifteen minutes and titrating with one-half normal hydrochloric acid, using a very dilute alcoholic solution of phenol-phthalein as an indicator.

According to Valenta, who tested a number of animal and vegetable fats by Koettstorfer's method, the following is the most suitable process: The fat to be examined (if solid) is first melted and then filtered; 1 to 2 grammes are then brought into a wide-necked flask, closed during the subsequent heating by means of a funnel inserted in the neck of the flask.

The standard (titer) of alcoholic potash lye changes very rapidly; it is therefore recommended to test it before every series of experiments. This is done by measuring out 25 cubic centimetres by means of a pipette, then heating ten to fifteen minutes in a water-bath, and finally titrating with half normal hydrochloric acid, using alcoholic solution of phenol-phthalein as an indicator. The lye is prepared by dissolving 28.05 grammes of caustic potash in 96 per cent. alcohol, and increasing the saturated cold solution to 1 liter by the addition of alcohol.

For the determination of the saponification equivalent, the potash lye is brought to 59° F.; 25 cubic centimetres are then

measured off by means of a pipette and added to the quantity of fat in the flask. The whole is then heated in a water-bath kept near the boiling temperature. Heating for ten to fifteen minutes generally suffices, though some fats require a longer time; for cocoanut-oil it is, however, recommended not to exceed twelve minutes in order to obtain useful results. The clear soap solution is compounded with phenol-phthalein, and the excess of potash titrated back with half normal hydrochloric acid, the point of neutrality being very sharply indicated by the yellow color of the liquid.

The final results are expressed by the number of milligrammes of potash (KHO) which saponify 1 gramme of fat.

According to the results obtained, Valenta divides the oils into three groups:—

1. Mean saponification number, 193.0: Oils of apricot, almond, peanut, cotton-seed, olive, and sesame.
2. Mean saponification number, 188.1: Oils of pumpkin-seed, olive kernels.
3. Mean saponification number, 177.1: Oils of colza, rape, hedge radish, and castor.

Very valuable for testing fats is also the iodine absorption process recommended by Baron Hübl. It is based upon the fact that the fatty acids can be divided into four groups: The acetic, acrylic, linoleic, and ricinoleic acids series. The relative proportion of these acids in a variety of fat may be assumed as constant within certain limits and differing only in different kinds of oil, but the members of the four groups of acids exhibit a very different behavior towards haloids. While under ordinary circumstances the first group is indifferent towards this series of bodies, the second group and ricinoleic acid readily unite with two atoms, and the third group with four atoms of a haloid. The members of the first group are, therefore, termed saturated fatty acids, and those of the second and third groups non-saturated. If, therefore, it is possible to unite a fat with a haloid under circumstances which exclude substitution, and it is also possible accurately to determine the added quantity of the haloid, the number thus obtained would be almost a constant for each fat, and would be dependent upon the kind and relative amount of non-saturated



acids present in the fat. And as, besides, there is a difference in the molecular number of the non-saturated fatty acids occurring in nature, the quantity of haloid added must also vary with different fats. Theoretically, it is claimed, the non-saturated acids occurring in the fats unite with the following quantities of iodine:—

Name of fatty acids.	Formula.		Number of double combinations.	100 grammes of the acids unite with iodine.
Hypogæic acid	$C_{16}H_{30}O_2$	. . .	1	100.00 grammes.
Oleic acid	$C_{18}H_{34}O_2$	. . .	1	90.07 “
Erucic acid	$C_{22}H_{42}O_2$	. . .	1	75.15 “
Ricinoleic acid	$C_{18}H_{34}O_3$	. . .	1	85.24 “
Linoleic acid	$C_{16}H_{28}O_2$	. . :	2	201.59 “

Of the haloids the use of iodine was for many reasons found more convenient and suitable than chlorine or bromine. Experiments, however, soon showed that at an ordinary temperature iodine acts very inertly upon fats, while at higher temperatures its effect is very unequal, and a smooth reaction in the above-indicated sense cannot be produced under these circumstances. An effect satisfactory in every respect is, however, produced by an alcoholic solution of iodine and mercuric chloride. This mixture reacts upon the non-saturated fatty acids at an ordinary temperature, products of chlorine and iodine combinations being formed, while the saturated acids present remain unaltered. The mixture acts in the same manner upon the free fatty acids, as upon glycerides, which, together with the easy volumetric determination of iodine, make this test a very simple one.

For the execution of the test the standard solution is prepared by dissolving 25 grammes of iodine and 30 grammes of mercuric chloride, each in one-half liter of 95 per cent. alcohol, and uniting the two solutions. After standing from six to twelve hours, the strength of the solution is determined by means of a standard solution of sodium hyposulphite.

The method of applying the test is as follows: Weigh out, as the case may be, 0.2 to 0.3 gramme of drying oil, 0.3 to 0.4 gramme of non-drying oil, and 0.8 to 1 gramme of solid fat, dissolve it in 10 cubic centimetres of chloroform, and add an excess of the above-standard iodine solution. After standing for one-and-a-half to two hours, determine the excess of iodine by the sodium hyposulphite solution. By calculation, the number of

grammes of iodine taken up by 100 grammes of oil is then found, and this number is the constant for the fat examined.

The following are some of the instances in which the quantity of iodine per 100 grammes of oil has been determined :—\*

						Iodine degree.	
						Hübl.	Möller.
Shark-liver oil	.	.	.	.	.	—	268.2
Menhaden-oil	.	.	.	.	.	—	170.8
Porpoise-oil	.	.	.	.	.	—	131.2
Seal-oil	.	.	.	.	.	—	103.4
Linseed-oil	.	.	.	.	.	158	175.7
Walnut-oil	.	.	.	.	.	143	—
Poppy-seed oil	.	.	.	.	.	136	—
Cotton-seed oil	.	.	.	.	.	105	107.9
Rape-seed oil	.	.	.	.	.	100	99.4
Almond-oil	.	.	.	.	.	98.4	—
Castor-oil	.	.	.	.	.	84.4	—
Olive-oil (a)	.	.	.	.	.	82.8	81.3
“ “ (b)	.	.	.	.	.	—	102.9
Lard-oil	.	.	.	.	.	59	47.2
Palm-oil	.	.	.	.	.	51.5	48.6
Tallow	.	.	.	.	.	40.	—
Cocoa-nut oil	.	.	.	.	.	8.9	6.8
Cocoa butter	.	.	.	.	.	—	34.4
Muskat butter	.	.	.	.	.	—	31.6
Mutton fat	.	.	.	.	.	—	57.3
Lard	.	.	.	.	.	—	55.0
Beef suet	.	.	.	.	.	—	38.4
Oleic acid	.	.	.	.	.	—	86.2
Olein	.	.	.	.	.	—	82.3
Commercial stearin	.	.	.	.	.	—	1.7
Beeswax	.	.	.	.	.	—	5.3
Japanese wax	.	.	.	.	.	4.2	5.61
Bayberry tallow	.	.	.	.	.	—	1.38
Butter	.	.	.	.	.	31	36.8
Oleomargarin	.	.	.	.	.	55.3	53.5
“ Butter” (a)	.	.	.	.	.	—	42.8
“ “ (b)	.	.	.	.	.	—	43.0
“ “ (c)	.	.	.	.	.	—	43.6
Oleomargarin, 43.71 per cent.	}	.	.	.	.	—	43.52
Butter, 56.29 “							
Oleomargarin, 19.54 per cent.	}	.	.	.	.	—	28.07
Butyric acid, 31.94 “							
Butter, 48.52 “	}	.	.	.	.	—	88.54
Olive-oil, 68.24 per cent.							
Cotton-seed oil, 31.76 “	}	.	.	.	.	—	54.36
Butter, 47.88 per cent.							
Oleomargarin, 33.29 “	}	.	.	.	.	—	
Cotton-seed oil, 18.82 “							

\* The butters marked (a), (b), and (c) contained 90 to 91 per cent. insoluble and 4.2 to 4.6 per cent. soluble fatty acids. “Olive-oil” (b), though sold for genuine oil, was apparently (by this test) mostly cotton-seed oil.

Other chemical reactions used in testing fats consist of color phenomena appearing under the action of certain chemicals. Mailho has, for instance, observed that all oils from Cruciferae (rape, colza, German sesame) form, when treated with aqueous potash or soda lye, potassium sulphide and sodium sulphide, but that this formation does not take place in other oils. He boiled 25 to 30 grammes of the oil with a solution of 2 grammes of pure sodium hydrate in 20 grammes of water and then filtered through a filter previously moistened. A strip of paper previously moistened with solution of sugar of lead or sugar becomes black if the oil is derived from Cruciferae or contains such in admixture. If the sample is boiled in a silver dish, the latter becomes black, even if the oil under examination contains but one per cent. of an oil derived from the Cruciferae. This manipulation is, however, liable to errors, since oils extracted with carbon bisulphide are generally not entirely free from sulphur combinations, and therefore give the same reaction. Further, it occurs sometimes that barrels used for transporting oils are rinsed with sulphurous lyes, and the oil, though pure, will show the same reaction. To avoid this error it suffices to wash the oil, before testing, with pure water, stirring it with a silver spatula, whereby the alkaline sulphide, which is soluble in water, blackens the silver. The washing is continued until the water shows no trace of sulphur. The oil can then be tested in the above manner.

Schneider's method of testing oils for adulteration with colza or rape-seed oils may be considered as a modification of Mailho's. He shakes the oil with double its volume of ether, adds 20 to 30 drops of a saturated solution of nitrate of silver, and observes the coloration of the oil. If it contains colza or rape-seed oil, it becomes brown to black.

Th. Chateau\* and C. Calvert† have compiled extensive tables of the chemical characteristics of the various fluid fats. They should, however, not be implicitly relied upon, as the color reactions produced by chemical agents vary according to the manner of obtaining the oils, their purification, and their age.

\* Th. Chateau. *Die Fette*. Leipzig, 1864.

† Dingler's Polytech. Jour. 132, S. 282.



*Color reactions of Chateau.*—The test is effected by placing 10 drops of the sample upon a large watch-crystal or in a small porcelain capsule, and adding from a dropping tube 5 drops of the reagent.

In the following the drying oils are given under (a), the non-drying oils under (b), and oils of animal origin under (c).

1. CALCIUM DISULPHIDE.\*—This reagent is prepared by boiling milk of lime and flowers of sulphur for half an hour and filtering. The reagent should only be used after standing a few days. It is added, drop by drop, to the oil, the whole stirred together by means of a glass rod, and observed whether the gold color of the mixture remains permanent or disappears.

*Permanent gold color:* (a) oils of linseed, poppy, nut; (b) oils of olive (fine), sweet almonds, colza, rape, sesame, cameline, cotton; (c) sheep's-foot oil, oleic acid (develops sulphuretted hydrogen), sperm-oil.

*Transient gold color:* (a) oils of white poppy, hemp (first blackish-green, then dirty green-yellow), castor; (b) oils of olive (ordinary table-oil), peanut, beechnut; (c) neat's-foot oil, horse-bone oil, whale, seal, fish, cod-liver, and ray oils.

2. ZINC CHLORIDE.—Syrup-thick solution obtained by saturating hydrochloric acid with pure zinc oxide and evaporating to the consistency of syrup.

*White or scarcely affected:* (a) oils of poppy, nut; (b) oils of sesame, sweet almonds (on heating); (c) neat's-foot oil, horse-bone oil (at the ordinary temperature), sperm-oil, whale-oil, cod-liver oil (at the ordinary temperature).

*Yellow, orange, flesh color, or dark brown:* (a) linseed-oil (yellow), castor-oil (yellow shading to rose-red); (b) oils of rape, peanut, beechnut (flesh color), cotton-seed (dark brown); (c) horse-bone oil (on heating yellow), whale-oil (on heating brown-yellow), oleic acid, train-oil, seal-oil (brown-red), ray-oil (red-yellow at an ordinary temperature).

*Greenish-yellow, green, bluish-green:* (a) linseed-oil; (b) oils of

\* Allen (Moniteur scient. 14, 724) has shown this reaction to be of little value, since the different behavior of the oils depends only on their greater or smaller content of acid.

colza, cameline, sweet almonds (at an ordinary temperature), olive; (c) cod-liver oil (on heating), ray-oil (on heating).

3. SULPHURIC ACID of 1.843 specific gravity; 3 to 4 drops to 10 to 15 drops of oil.

*Brown shades:* (a) linseed-oil (French and East Indian, the latter after stirring), nut oil (stirred); (b) oils of peanut, beech-nut (stirred), cotton-seed; (c) neat's-foot oil (stirred), horse-bone oil (stirred), oleic acid, train-oil (black-brown), seal-oil (dark dragon's-blood color), sperm-oil (red-brown), whale-oil (red-brown), cod-liver oil (violet-red to crimson, on stirring blue-violet, then dragon's-blood color), ray-oil (same as cod-liver oil).

*Dark yellow, reddish-yellow, orange-yellow:* (a) Indian linseed-oil (orange-yellow without stirring), poppy-oil (pale yellow and orange-yellow), castor oil (pale yellow, then reddish-yellow); (b) olive-oil (yellow, when stirred yellow or reddish-yellow), sesame-oil, beechnut-oil (dark yellow, without stirring), oil of sweet almonds (yellow, without stirring), cameline-oil (reddish-yellow); (c) neat's-foot oil (yellow, then orange), sheep's-foot oil (yellow and reddish-yellow).

*Green veins, or, on stirring, green or greenish colorations:* (a) hemp-seed oil, linseed-oil (English, stirred); (b) oils of colza, cameline, olive (virgin-oil, lamp-oil), sesame (stirred), rape, sweet almonds (stirred, yellowish-green), cameline (green veins); (c) none.

4. STANNIC CHLORIDE.—The fuming perchloride of tin of commerce.

*a. Immediate coloration:—*

*Yellow or colorless:* (a) oils of poppy and castor; (b) oils of olive (fine), sesame (pale yellow), sweet almonds (no coloration); (c) neat's-foot oil, sheep's-foot oil (pale yellow).

*Brown-red, pale-brown, reddish-yellow, brown-yellow:* (a) oils of linseed (brown-yellow), poppy (reddish-yellow), walnut (reddish-yellow); (b) olive-oil (oil of the infernal regions, reddish-yellow), peanut-oil (pale brown), cameline-oil, beechnut-oil (reddish-yellow), cotton-seed oil (orange-yellow); (c) neat's-foot oil and horse-bone oil (reddish-yellow), whale-oil (orange), sperm-oil (red-brown shading into violet), seal-oil, train-oil (dark red-brown).

*Green:* (a) linseed-oil (green veins or bluish-green), hemp-oil (green); (b) olive-oil (lamp-oil), rape-oil, colza-oil; (c) cod-liver oil (violet-blue, red-violet, crimson, dragon's blood color), ray-oil (same as cod-liver oil).

*b. Final result.—Color of the thickened or solidified mass.*

*Yellow:* (a) oils of poppy, castor (pale yellow); (b) oils of olive, sesame, sweet almonds, cameline (pale yellow); (c) sheep's-foot oil (pale reddish-yellow).

*Brown, pale-brown red, orange, red-yellow:* (a) linseed-oil (pale orange, brown-gray, red-yellow); (b) olive oil (ordinary, orange-yellow), colza-oil, peanut-oil (brown-red), beechnut-oil (pale red-yellow), cotton-seed oil (yellow-brown); (c) neat's-foot oil (orange-yellow), horse-bone oil (orange-yellow), oleic acid (does not solidify, red-brown), whale-oil (pale mahogany color), sperm-oil (orange-yellow), seal-oil (dark-brown red), train-oil (dark-sepia brown), cod-liver oil (dark orange), ray-oil (dark orange).

*Green:* (a) hemp-oil (dark green); (b) olive-oil (lamp-oil, dirty green), rape-oil (dirty green); (c) ray-oil (greenish-yellow shading into reddish).

5. SYRUPY PHOSPHORIC ACID, *i. e.*, ordinary phosphoric acid evaporated to 1.72 specific gravity.

*a. Cold; stir and observe—*

*White, no coloration, decoloration, gray, slightly yellowish:* (a) oils of poppy, nut, castor (all white); (b) oils of sweet almonds, rape, cameline (all decolorized); (c) neat's-foot oil and sheep's-foot oil.

*Yellow, orange-yellow:* (a) linseed-oil; (b) sesame-oil (straw and orange-yellow), peanut-oil (straw color), cotton-seed oil (gold yellow); (c) whale-oil (straw, then orange-yellow), neat's-foot oil, horse-bone oil (orange-yellow), oleic acid (straw color), sperm-oil (straw color), seal-oil (pale brown-red), train-oil (red-yellow), cod-liver oil (red-yellow), ray-oil (gold-yellow).

*Green:* (a) linseed-oil, hemp-oil (dark green); (b) oils of olive, colza, rape, cameline.

*b. Heat; and observe—*

*Colorless:* (a) poppy oil; (b) olive oil (fine); (c) sheep's-foot oil.

*Yellow to orange-yellow:* (a) linseed-oil (pale yellow), poppy-oil



(pale yellow), hemp-oil (reddish-yellow), nut-oil (pale yellow), castor-oil (pale yellow); (b) olive-oil (ordinary, reddish-yellow), oil of sweet almonds (pale yellow), colza-oil (pale yellow) peanut-oil (gold-yellow), cameline oil (pale yellow), sesame-oil (straw color), beechnut-oil (straw color), cotton-seed oil (reddish-yellow); (c) neat's-foot oil (pale yellow and gold-yellow), horse-bone oil (gold-yellow), oleic acid (gold-yellow), sperm-oil (pale yellow).

*Brown:* (c) seal-oil (black-brown), train-oil (black-brown), whale-oil (red-brown), cod-liver oil (blackish-green), ray-oil (red-brown).

*Froth, gray or white, brown, black or blackish:* (a) linseed-oil (gray or blackish), poppy-oil (gray), hemp-oil (gray and greenish), castor-oil (white); (b) sesame-oil (greenish), olive-oil (ordinary and oil of the infernal regions, gray), colza-oil (brown), rape-oil (brown), peanut-oil (gray), beechnut-oil (brown), cotton-seed oil (gray); (c) horse-bone oil (blackish), seal-oil, train-oil, and whale-oil (all greenish-black), sperm-oil (gray), cod-liver oil (dirty greenish-gray), ray-oil (same as cod-liver oil).

6. *Mercuric nitrate*, prepared by dissolving mercury in an excess of pure nitric acid. First observe the effect given by the salt alone, and then add two or three drops of sulphuric acid and observe the color of the liquid which covers the precipitate.

*a. Colors given by the salt alone.*

*White, gray, or colorless emulsion:* (a) poppy-oil (white, shading slightly into yellowish), nut-oil (no coloration), castor-oil (white); (b) oil of sweet almonds (grayish-white), sesame-oil (white), beech-nut oil (colorless); (c) neat's-foot oil, sheep's-foot oil, oleic acid, and sperm-oil (colorless).

*Yellow:* (a) linseed oil (straw color, or pale yellow with dark-yellow veins); (b) olive-oil (oil of the infernal regions, canary-yellow), rape-oil (straw color), peanut-oil (pale yellow), cameline-oil (straw color), sesame-oil (orange-yellow), cotton-seed oil (pale yellow); (c) neat's-foot oil (red-yellow), horse-bone oil (orange yellow), whale-oil (straw color), seal-oil (reddish-yellow), train-oil (gold-yellow), cod-liver oil (straw color), ray-oil (pale yellow).

*Green:* (a) linseed-oil, hemp-oil (greenish after stirring); (b) olive-oil (greenish-yellow), colza-oil, rape-oil (sea-green), cameline-oil (pale green); (c) none.

*b. Color of the liquid above the precipitate, by sulphuric acid added after mercury nitrate.*

*Gray, rose-red:* (a) hemp-oil (stirred, greenish-gray); (b) colza-oil (dirty flesh-color, then reddish-gray), rape-oil (brownish-gray); (c) sheep's-foot oil (flesh-red).

*Yellow, orange yellow:* (a) linseed-oil (dirty yellow, dark yellow, reddish-yellow), poppy-oil (reddish-yellow), castor-oil (first gold-yellow, then canary-yellow); (b) olive-oil (ordinary and oil of infernal regions, reddish-yellow), sesame-oil (green veins, then orange-yellow); (c) neat's-foot oil (reddish-yellow), horse-bone oil (dirty yellow-brown).

*Brown:* (a) linseed-oil (sepia-brown, then dirty yellow red-brown), poppy-oil (dark brown), nut-oil (pale brown to black-brown), castor-oil (dark brown), hemp-oil (not stirred, dark red-brown); (b) olive-oil (best quality, sepia-brown shading into gray), olive-oil (lamp-oil, reddish-brown), oil of sweet almonds (pale chocolate-brown), colza-oil (brownish rose-red, then pale brown), peanut-oil (chocolate-brown), cameline-oil (red-brown, then chocolate-brown), beechnut-oil (pale red-brown), cottonseed-oil (pale chocolate-brown); (c) neat's-foot oil (chocolate-brown, red-brown), horse-bone oil (same as neat's-foot oil), oleic acid (pale chocolate-brown), whale-oil (dark chocolate-brown), sperm-oil (pale to dark brown), seal-oil (black-brown), train-oil (black-brown), cod-liver oil (dark brown), ray-oil (sepia-brown).

*Sudden effervescence, development of vapors of nitrous acid:* (a) linseed-oil, nut-oil, castor-oil; (c) tallow-oil, seal-oil. None of the other oils effervesce when treated in this manner.

By Crace-Calvert's method, which is much used, the changes of colors are observed, which the oils undergo on treatment with sulphuric acid and nitric acid of determined concentration, with phosphoric acid and nitro-muriatic acid; and further the changes in color and consistency which occur on boiling the fresh oil or that treated with nitric acid or nitro-muriatic acid with soda lye.

For all the reactions 5 volumes of oil are mixed with 1 volume of acid (or soda lye), and the mixture after stirring allowed to stand five to fifteen minutes, according to the degree of concentration of the acids. For treating the oils already mixed with

acids with soda lye, 10 volumes of lye to 5 volumes of oil are taken.

The following are used:—

1. *Soda lye of 1.340 specific gravity.*—The mixture is heated to boiling. Characteristic for train-oil (dark-red coloration). For distinguishing hemp-oil (brown-yellow, solid) and linseed-oil (yellow, liquid).

2. *Sulphuric acid of 1.475 specific gravity.*—Allow the mixture to stand for fifteen minutes; characteristic for hemp-oil and linseed-oil (green coloration), and for train-oil (red coloration).

3. *Sulphuric acid of 1.530 specific gravity.*—Observe the coloration after five minutes.

4. *Sulphuric acid of 1.635 specific gravity.*—Observe after five minutes.

5. *Nitric acid of 1.180 specific gravity.*—Observe after five minutes. For the distinction of hemp and linseed oils.

6. *Nitric acid of 1.220 specific gravity.*—Characteristic for oils of nut, sesame, hemp, and poppy.

7. *Nitric acid of 1.330 specific gravity.*—Very characteristic for oils of nut and sesame.

8. The mass obtained according to 7 is heated to boiling with *soda lye of 1.340 specific gravity.*

9. *Syrupy phosphoric acid (with 3 equivalents of water).*—Very characteristic for train-oil.

10. *1 volume of nitric acid of 1.330, and 1 volume of sulphuric acid of 1.345 specific gravity.*—Characteristic for sesame oil.

11. *Nitro-muriatic acid.*—25 volumes of hydrochloric acid and 1 volume of nitric acid of 1.330 specific gravity.

12. The mass obtained according to 11 is treated with soda lye.

The accompanying table shows the reactions of oils according to Crace-Calvert.





*Foreign admixtures* with which the fats or oils may be contaminated or adulterated are determined with greater ease than additions of other fats or oils. The most frequent contaminations are *water* and *sand* (dirt). To determine the first, heat 40 to 50 grammes of the fat under examination in a tared beaker glass at  $230^{\circ}$  F., first for one hour with occasional stirring, and then for two hours, without stirring, at  $247^{\circ}$  F. The difference in weight shown by the fat thus dried as compared with the original sample is the content of water.

To determine the content of dirt, melt the dried fat in a beaker glass, then filter through a tared filter, wash the latter with hot benzene and dry it at  $176^{\circ}$  to  $194^{\circ}$  F. The increase in weight of the filter gives the dirt contained in the fat. Any moisture of the filter, after drying at the above temperature, is due to a small amount of glycerin. In this case the filter is extracted with alcohol and again dried.

In consequence of incomplete purification after refining, the fats may contain *sulphuric acid*, *alkaline carbonates*, *alum*, and *lead*. *Sulphuric acid* is found by vigorous shaking of the oil with distilled water, and, after allowing the aqueous fluid to settle, compounding it with barium chloride. A white precipitate indicates the presence of sulphuric acid. *Alkaline carbonates* are determined by shaking the oil with water and testing the latter for alkaline reaction with litmus paper. The presence of alum is shown by shaking the oil with water containing some nitric acid, evaporating the aqueous solution, and compounding with ammonia. A white precipitate proves the presence of alum.

The fat oils are frequently adulterated with cheaper fat oils, principally *resin-oil* and *mineral oils*. The most reliable method of showing their presence is the saponification process first recommended by Thompson, *i. e.*, saponification of the oil under examination, mixing the soap formed with sand, drying the soap, and extracting with petroleum ether. The method proposed to shake the liquid soap directly with petroleum ether and to separate the latter from the soap solution by means of a separatory funnel, cannot be recommended. Finkener\* declares the last method entirely useless when the oil contains less than 10 per cent. of

\* Mittheilungen aus der Koenigl. techn. Versuchsanstalt, Berlin, 1886, S. 13.

non-saponifiable oil, and uses alcoholic soda lye for saponification, while Thompson recommended alcoholic potash lye. Finkener prefers soda, because potash-soap is more difficult to dry than soda-soap, and proceeds as follows:—

Dissolve 35 grammes of *Natrium hydricum purum* in 85 cubic centimetres of water and pour the hot solution into 730 grammes of boiling hot alcohol. Bring 10 grammes of the oil to be examined into a flask of 300 cubic centimetres' capacity, add 50 cubic centimetres of the alcoholic soda solution, boil the whole for 15 minutes in a water-bath and compound with 5 grammes of sodium bicarbonate for the conversion of the excess of soda used into carbonate. Pour the solution upon 200 grammes of pure dry sand in a metal evaporating dish and heat in a water-bath, with constant stirring, until the odor of alcohol has entirely disappeared. The warm mass is then brought into a glass cylinder of 500 cubic centimetres' capacity and provided with a stopper, and, after cooling, 300 cubic centimetres of petroleum ether, with a boiling point below 212° F., are added and the whole is thoroughly shaken for some time. The ether is then filtered off through a dry filter into a dry flask and 150 cubic centimetres of the filtrate are distilled in a still. The residue from the still is brought with a little petroleum ether upon a watch-crystal and dried in a water-bath until the petroleum ether disappears.

In examining commercial oils in the manner described, 0.5 to 3 per cent. of non-saponifiable residue is obtained even if there be no reason for suspecting adulteration. An intentional addition of mineral-oil can, therefore, be only assumed when more than 5 per cent. of non-saponifiable matter is found.

Whether the residue consists of resin-oil or mineral-oil is recognized by treating it with a mixture of 10 volumes of alcohol of 0.8182 specific gravity at 60° F. and 1 volume of chloroform. Resin oils dissolve, according to Finkener's\* investigations, at 73.5° F. by shaking with 10 times their volume of this mixture, while mineral oils with a high boiling point do not dissolve even if shaken with 100 times their volume. Demski and Morawski†

\* Mittheilungen aus der Koenigl. techn. Versuchsanstalt, Berlin, 1885, S. 160.

† Dingler's Polytechn. Jour. 258, S. 39.



treat the oily residue with acetone. If it dissolves in an equal volume of acetone, resin-oil, or resin-oil compounded with a little mineral-oil, is indicated; if a portion remains undissolved, the sample consists of mineral-oil or mineral-oil compounded with a little resin-oil.

If the quantity of non-saponifiable oil in a fat amounts to more than 10 per cent., Finkener seeks to establish the fact by the determination of the alkali required for saponification, therefore by simple titration. "The natural fluid fats contain over 50 per cent. of olein, and as palmitin, olein, and stearin require 20.84 per cent., 19.00 per cent., and 18.87 per cent. of potassium hydroxide for saponification, an average of about 19 per cent. will be necessary. To test the oils in this respect, dilute sulphuric acid (124.2 grammes of  $\text{SO}_2$  to the litre), an alcoholic solution of potash of which 50 cubic centimetres are equivalent to 13 cubic centimetres of sulphuric acid, and a solution of phenol-phthalein in 100 parts of alcohol are used. The potash solution is prepared by dissolving 55 parts by weight of *Kalium hydricum purum* in 85 parts by weight of water and pouring the hot solution into 730 parts by weight of boiling hot absolute alcohol. The next day the clear solution is drawn off from the sediment and diluted with 90 per cent. alcohol to the desired concentration. Ten grammes of the oil, weighed in an Erlenmeyer flask of 300 cubic centimetres' capacity, are boiled with 50 cubic centimetres of potash solution for 15 minutes, with frequent shaking for the first few minutes. Saponification proceeds in this manner without jolting of the fluid.

"After adding 5 drops of phenol-phthalein, the liquid is titrated with sulphuric acid to somewhat above the point of decoloration, the carbonic acid expelled by boiling, and the red coloration restored by titration with the potash solution. The recognition of the change of color of the phenol-phthalein presents no difficulty, even if, in the presence of dark-colored mineral oils, the saponified sample assumes a brown color."

This simple method would be an excellent one if the fats and fat oils consisted only of palmitin, stearin, and olein. But we have a number of fats with a saponification equivalent materially differing from that assumed above, especially cocoanut-oil and

palm-kernel oil, which, on account of their percentage of lauro-stearin, fix a considerably larger quantity of potassium hydrate, and the oils from the *cruciferae*, which, on account of their percentage of erucic acid, fix a smaller quantity. Finkener's method is, therefore, available only for a limited number of oils.

The presence of *resin* in fats is readily shown by its solubility in alcohol and soda solution. By repeatedly heating the sample of fat with 70 per cent. alcohol, the resin passes into solution. After precipitating it with water, the precipitate is concentrated by heating, and, if necessary, by the addition of some hydrochloric acid; it can then be readily recognized as resin by its appearance, odor, etc. Barfoed heats the fat with a soda solution prepared by dissolving 1 part of crystallized soda in 3 parts of water and adding 7 parts of 30 per cent. alcohol (2 volumes of alcohol of 93 per cent. and 5 volumes of water). The resin passes into solution and is separated by acidulating and heating.

As the commercial fats contain more or less free fatty acids, the above methods are not available for the quantitative determination of resin. For this purpose the fat must first be saponified and the quantity of resin determined as described later on for the determination of resin in soaps.

The presence of *free fatty acids* in oils can be readily detected. A very simple method is given by Wiederhold.\* By pouring the oil to be examined over cuprous oxide in a white glass, the layer next to the cuprous oxide assumes a green color if the oil contains acid. The appearance of the reaction is promoted by moderate heating. To test the neutrality of the oils Allaire† shakes them with a solution of sodium bicarbonate in water. If the oil separates in shining globules it is neutral, but if it becomes turbid and a partial saponification takes place it contains free fatty acids.

For the quantitative determination of fatty acids, Mayer dissolves 2 to 3 grammes of fat in 20 cubic centimetres of ether, compounds the solution with 10 cubic centimetres of alcohol and some solution of phenol-phtalein, and adds, according to the con-

\* Dingler's Polytechn. Jour., 217, 314.

† Octave Allaire, Notice sur les huiles neutres raffinées, p. 11.

tent of acid,  $\frac{1}{10}$  or  $\frac{1}{1}$  normal lye, until the liquid assumes a red coloration. Aqueous potash lye is preferable to the alcoholic, on account of the constancy of its titer, which constantly changes in the alcoholic solution, and must, therefore, always be determined anew before each series of experiments; on the other hand, the fat readily separates from its alcoholic solution by the addition of aqueous lye, and has then again to be brought into solution by heating in a water-bath. With an alcoholic-ethereal solution two layers are formed, and the reaction must be carried on to the end with vigorous shaking after each fresh addition of lye.\*

Titration does not give the per cent. by weight of the free fatty acids contained in the fat, but only the quantity of alkali required for the neutralization of a determined weight of the sample. For oils this is generally satisfactory, the number of cubic centimetres of normal lye required for the neutralization of 100 cubic centimetres of the sample being given.

The quantity of *glycerin* contained in a fat is determined in the usual manner, by saponifying the fat with alcoholic potash or soda lye, expelling the alcohol by evaporation, dissolving the soap in water, adding dilute sulphuric acid and boiling moderately until the fatty acids have become entirely clear. The mass is then allowed to cool, and, after filtering the liquid containing glycerin from the congealed fatty acids, the latter are once more boiled in water, allowed to congeal, and the filtered wash-water is united with the first filtrate. The latter is exactly neutralized with sodium carbonate and then evaporated to dryness in a water-bath. The residue consisting of sodium sulphate and glycerin is treated with alcohol, which leaves the sodium sulphate undissolved. The filtered alcoholic solution is evaporated, the residue again treated with alcohol, and the filtered solution evaporated in a platinum dish in a water-bath.

On account of the volatility of glycerin at  $212^{\circ}$  F., the numbers obtained by the foregoing process are always too low. The most reliable process, and one which is available for all cases, is

\* Benedikt. *Analyse der Fette u. Wachsarten*, S. 93.



the determination of the glycerin by oxidation with potassium permanganate.

When glycerin in a strongly alkaline solution is oxidized at an ordinary temperature with potassium permanganate, 1 molecule of glycerin yields quantitatively exactly 1 molecule each of oxalic and carbonic acid—



Upon this is based the determination of glycerin by Benedikt and Zsigmondi,\* the principle having been first established by Fox.

The fat is saponified with calcium hydrate and entirely pure methyl alcohol, the residue dissolved in hot water, and the soap decomposed with dilute hydrochloric acid. It is then heated until the fatty acids have separated clear. With fluid fats it is advisable to add some hard paraffin in order to congeal the fatty acids floating on top during the subsequent cooling, which is effected by placing the dish in cold water. The fluid is then filtered into a capacious flask, neutralized with potash lye, and 10 grammes of caustic potash are added. 5 per cent. solution of potassium permanganate is then added at an ordinary temperature until the fluid is no longer green, but assumes a blue or blackish coloration. It is then heated to the boiling point, whereby manganese peroxide is separated and the fluid becomes red; as much aqueous sulphurous acid is then added as is required for complete decoloration. It is then filtered through a smooth filter of sufficient size to receive at least one-half of the entire fluid, and thoroughly washed with boiling water. The last washwaters are frequently rendered turbid by some manganese peroxide; this turbidity disappears, however, by the action of the sulphurous acid liberated during the subsequent acidulation with acetic acid. The fluid is then heated to nearly the boiling point and precipitated with calcium chloride or calcium acetate. As, besides calcium oxalate, the precipitate contains always some silicic acid and frequently gypsum, it cannot be considered, after calcining, as pure calcium carbonate, nor as calcium oxide. The determination of the calcium oxalate contained therein is

\* Chemiker Zeitung, 9, S. 975.

best effected by titration, either with potassium permanganate in acid solution or by alkalimetry after calcining. For the latter purpose the calcined precipitate is dissolved in about half-normal solution of hydrochloric acid, titrated back with about half-normal soda lye with an addition of dimethyl-aniline orange as an indicator. The titer of the hydrochloric acid is generally fixed for sodium carbonate; 106 parts of sodium carbonate correspond to 92 parts of glycerin.

In regard to the above process we would remark that methyl alcohol is used for the saponification of the fats instead of ethyl alcohol, because the latter, at certain concentrations and with a determined percentage of alkali in the solution, is converted by potassium permanganate into oxalic acid. This creates errors which are the greater the more alcohol is retained by the soap in drying. By repeated evaporation and renewing of the water to expel the last traces of alcohol, a portion of the glycerin would also be lost.

The fluid subjected to oxidation contains besides glycerin all the soluble fatty acids; by oxidation with potassium permanganate, according to the above process, they yield, however, neither oxalic acid nor another acid which can be precipitated by lime in acetic acid solution, so that their presence does not influence the determination of the glycerin.

Whether an oil obtained by extraction is free from carbon bisulphide can, according to O. Braun, be readily recognized by stirring about 30 parts by weight of lye of 40° B. under 60 parts by weight of oil, and allowing the soap formed to stand in a warm place for one hour. If the oil is badly purified, the soap is dark green and of a bad odor, which, however, disappears in time (weeks or months), leaving the soap externally faultless.

## CHAPTER V.

FATS, FAT OILS, FATTY ACIDS, AND RESIN USED IN THE  
FABRICATION OF SOAP.

ACCORDING to their derivation, the fats are generally divided into animal and vegetable fats and oils. We commence our descriptions with the

*Fats of Animal Origin.*

*Tallows.*—Under this collective term are understood the masses of fat found in abundance in the abdominal cavity, around the kidneys, etc., of the ruminantia, especially of those which have been fattened. In commerce a distinction is made between *beef tallow*, derived from oxen, cows, and calves, and *mutton tallow*, from sheep and goats, the latter being firmer and whiter, though otherwise there is no material difference. Besides, the firmness of the tallow from the same variety of animals is not always alike; it depends on the race, age, and especially on the food of the animal. Animals fed upon dry food furnish the most solid tallow, that of those pastured being less so, and that of those fed upon swill very soft.

The crude tallow as furnished by the butchers is enveloped in very thin cellular tissues and more or less contaminated with particles of skin, blood, etc. If kept for several days, these particles become decomposed and putrefy. It is necessary, therefore, especially in summer, to keep the tallow in a cool place or at once to separate it from the membranes by rendering.

Rendering is effected either by the *dry* or the *wet* process. For the first the tallow is cut up and the membranes enveloping it torn asunder by the application of heat; by the second process the tallow is boiled with dilute acids or alkalies which dissolve the cell substance.



Dry rendering is the older method. The fatty tissues are cut into small cubes, placed in a copper over an open fire, and exposed to a heat exceeding that of boiling water. A small percentage of water is frequently added, especially in summer, when the tallow has lost much of its natural moisture by evaporation. In the heat the membranes are destroyed and the melted tallow runs out, the membranous substances collecting on the top. These are removed and pressed so as to free them from fat. The solid matters—now called greaves or cracklings—form flat cakes and are sold as food for dogs, manure, or for use in the manufacture of lubricants and of ferrocyanide of potash. The melted fat is passed through a sieve into another suitable vessel and washed with boiling water. The impurities settle down with the water and the fat is drawn off into tubs and allowed to cool. This process has been in practice for a long time, and, notwithstanding its many inconveniences, is so still, especially in small establishments. Sometimes a still higher temperature is applied in order to cause the residuum to undergo a roasting, thereby trying to obtain a greater yield of pure tallow. In general, though, this method remains imperfect, and a larger or smaller loss of tallow is sustained, much remaining in the tissues, which are but imperfectly opened by this operation, and become so hard that they yield the tallow under the press with difficulty. Besides, it is an impossibility to obtain an even temperature in the copper, it becoming too high on the bottom to the detriment of the color and quality of the tallow. And furthermore the odor of the gaseous and other vapors developed from the animal substances during melting is so disgusting that from a hygienic standpoint this method of rendering has to be condemned.

This disagreeable odor is more readily removed by the application of steam in lieu of an open fire, but it is only a slight improvement in other respects, because the temperature remains too low, and besides, by the immediate contact of steam with the fat, the membrane is converted into glue, from which the tallow can be separated only with great difficulty. For this reason Appert proposed to render the tallow with one-third of its weight of water at  $239^{\circ}$  to  $266^{\circ}$  F., but even by this method the tallow is

not completely rendered out, especially if the raw material is not sufficiently comminuted.

By destroying the cells enveloping the fat as much as possible before heating, the rendering process is effected at quite a low temperature, the fat and membranous particles separating readily at 212° F. Several mechanical contrivances have been constructed for this purpose and are used in the manufacture of margarin, it being absolutely necessary in this branch of industry to obtain a tallow as pure as possible and uninjured by the action of a higher temperature; frequently the tallow is rendered at less than 212° F.

The destruction of the cells enveloping the tallow has also been attempted by the addition of chemical agents, this method having the further advantage that the odorous substances combine with the chemical agent, or are destroyed. D'Arcet was the first to recommend dilute sulphuric acid for this purpose. Bring 50 pounds of dilute sulphuric acid into the copper, then add gradually 1000 pounds of the comminuted fat divided into four equal portions, and finally 150 pounds of water previously compounded with 5 pounds of sulphuric acid of 60° B. The mass is then heated. By the action of the sulphuric acid, which partly dissolves and partly decomposes the membranes, the process of rendering even large quantities of tallow is finished in 1½ to 2½ hours. D'Arcet originally proposed the addition of acid for rendering over an open fire, but it is also available for steam rendering, which is now employed in all large establishments. In most rendering establishments, not working with a closed apparatus, open wooden vats lined with lead, and direct steam are employed. For 100 pounds of tallow 20 pounds of water and 1 pound of sulphuric acid of 66° B. are used, and steam of a pressure of 1 to 2 atmospheres is introduced. Another very suitable process consists in pouring sulphuric acid of 4° to 5° B. over the crude tallow as delivered by the butchers and loading it with boards and stones so that the sulphuric acid stands over the fat. After four or five days the acid is drawn off through a faucet in the bottom of the vat. The tallow is then rendered by the introduction of direct steam, the process being quickly accomplished, since

the cells have been partly destroyed by the acid. The greaves, which still contain tallow, are again acidulated and rendered out.

Evrard has proposed to mix and warm 300 parts of comminuted tallow with caustic soda lye (made of 1 part of calcined soda dissolved in 200 parts of water). The odorous substances combine with the soda and remain in the lye dissolved, while the pure fat is separated. Though the Société d'Encouragement of Paris examined this process and declared it an improvement, we prefer rendering with sulphuric acid.

Many attempts have been made to render innocuous the disagreeable vapors developed during rendering. The simplest method is to conduct them into a boiler fire. The idea originated with d'Arcet, who, in 1834, applied it to dry rendering. For this purpose it is, however, but little adapted, since the closed apparatus required impedes the necessary stirring; but it can be utilized in steam rendering. The most thorough investigations in reference to the destruction of the developing fetid odor were made by Grodhaus and Fink. The melting vat was provided with a well-fitting cover. In this cover was a hole three inches wide over which a tin pipe was fixed and carried to a contiguous boiler fire. The contents of the melting vat consisted of crude tallow, first and second quality mixed, and the requisite quantity of sulphuric acid. As the vapors developed in the melting vat they were easily drawn through the tin pipe into the flames without extinguishing the fire. They left the opening of the chimney without the least smell, proving that the fetid-smelling products had been destroyed. Experiments were also made to conduct the vapors under the grate of the fire-place, but it became very soon manifest that they extinguished the fire, so that this experiment was abandoned.

Among the many new inventions for rendering tallow, etc., over an open fire in a manner to avoid the offensive odors arising from the operation, that invented by Vohl is perhaps the most successful. Fig. 17 represents this apparatus. It consists of the cast-iron caldron *A*, lined with lead, the cylindrical head-piece *B*, and the cover *C*, with the mica plate *D*; another mica plate is in the door *E*, both serving for observing the processes in the

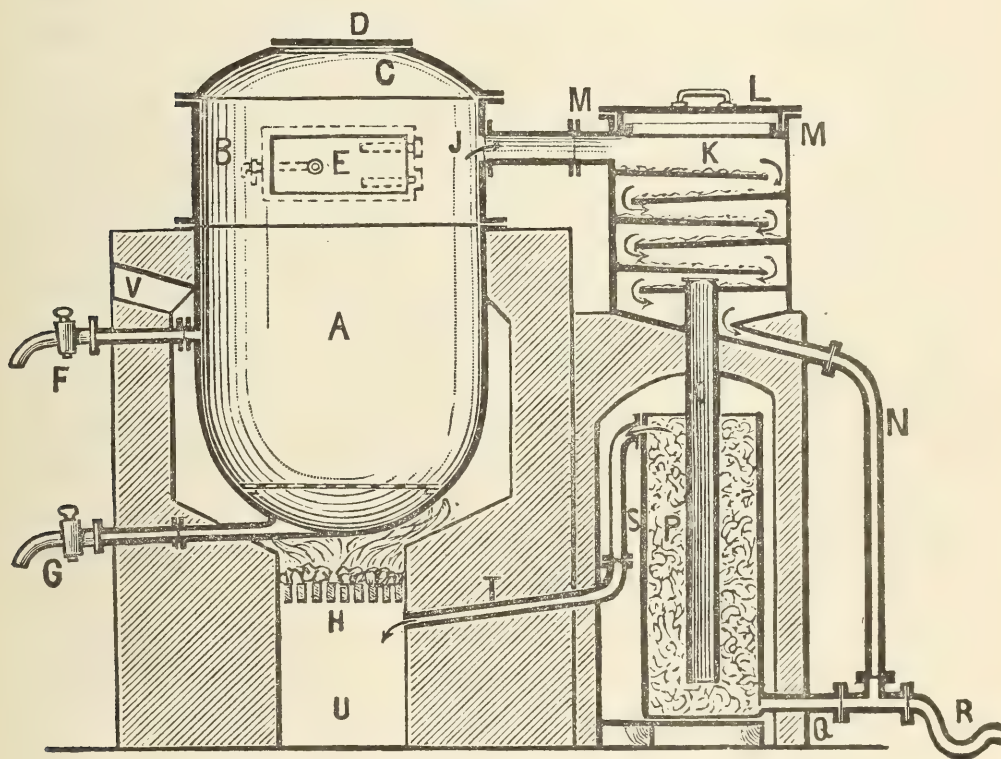


interior of *A*. At night a light is placed over the mica plate in the lid.

The tallow is introduced through the door *E*, which also serves to close hermetically the cast-iron head-piece *B*. After rendering is finished the tallow is discharged through the faucet *F* and the acid liquid through the faucet *G*, while the greaves remaining upon a perforated plate in the caldron are removed through the door *E*.

The gases and vapors developed during rendering in *A* pass through the pipe *J* into the condensing box *K*. The latter is closed with the cover *L*, provided with sand-joints at *M*.

Fig. 17.



Vohl's Rendering Apparatus over an open fire.

In the interior of *K* are oblique scaffolds covered with decomposed lime. The box itself is of wood saturated with asphaltum or tar. The water condensed in it runs off through the pipe *N*, the escape of gases being prevented by the curvature of *R* forming a trap.

The gases and vapors not condensed in *K* pass through the

pipe *Q* into the condenser *P*, which is lined with lead and filled with coke or pumice-stone saturated with sulphuric acid. The liquid collected here passes through the pipe *Q* to the discharge-pipe *R*.

The non-condensed gases, etc., are finally conducted through the pipe *S* into the channel *T*, which leads into the ash-pit *U* under the grate of the fire-place *H*.

The non-condensed gases, etc., being almost completely freed from aqueous vapors by passing through the condensers, can be unhesitatingly admitted under the grate without fear of disturbing the draught.

The ash-pit *U* is closed with an iron door, whereby a strong draught is produced which sucks all the gases from the apparatus under the grate. The gases of combustion escape to the flue *V*, which leads to the chimney.

An apparatus for rendering tallow by steam, invented, if we are not mistaken, by Lockwood & Everett, of New York, is shown in Fig. 18. Its principal advantage consists in the complete destruction of the noxious vapors and in the safety from explosions, as the fat in the digester melts very gradually.

The apparatus consists of two parts, a boiler or digester and a furnace for burning the developed gases and vapors, both being connected by the pipe *J*. The digester, which receives the fat to be rendered, consists of the steam-tight cylindrical boiler *A*, surrounded by the jacket *B*.

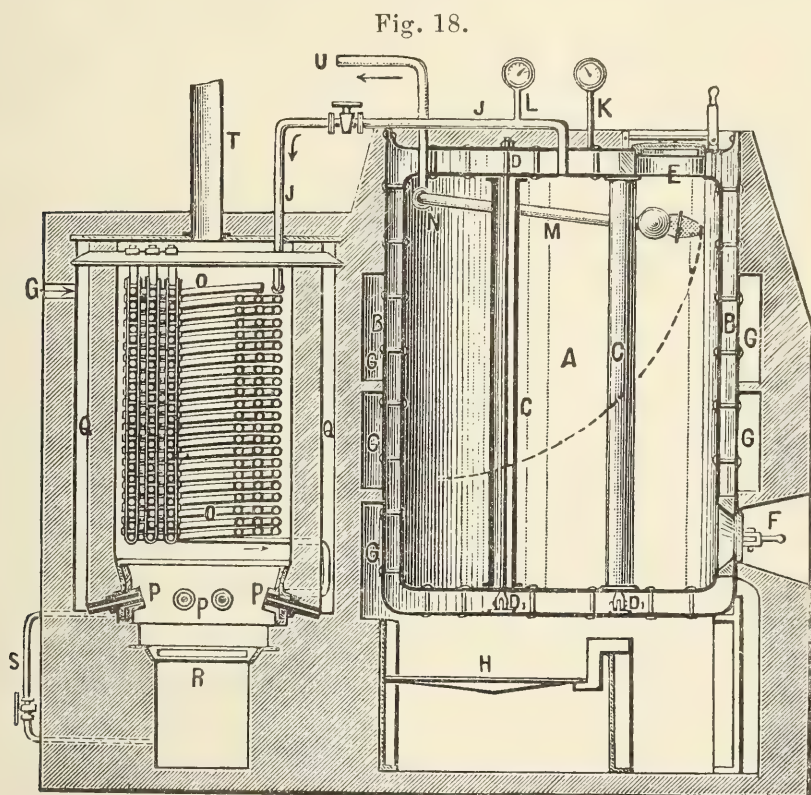
To secure greater solidity, as the digester must stand a hydraulic pressure of seven atmospheres, the bottoms are connected with the inner walls by the rods *D* and the jacket by stays. The steam-pipes *C*, which serve for uniformly heating the fat, contribute also to the strengthening of the digester.

Underneath the digester, which rests upon supports, is a fire-place from which the gases of combustion, after passing through the flues *G* in the brickwork over as large a surface of the digester as possible, escape into the chimney. The digester is filled through the manhole *E*, and the membranous residue removed through the opening *F*.

The pipe *M*, turning in the swivel-joint *N*, and provided on the end with a strainer to prevent the escape of foreign substances,

serves for the removal of the liquid fat. With the pipe *M* communicates the discharge-pipe *U*, by which the liquid fat can be conveyed to any desired place by the pressure prevailing in the digester *A*.

The gases and vapors developed in rendering escape through the pipe *J* into an Argand furnace, so called by the inventors.



Steam-Rendering Apparatus.

The gases pass here first through the heated pipe system *O*, and enter from below four burners, *P*, symmetrically arranged in a circle, where they are mixed with atmospheric air, ignited, and burned.

The gases of combustion, in ascending, pass around the spirals *O* and escape through the chimney *T*.

The air required for combustion enters at *Q*, in the upper part of the furnace, an air-chamber in the brickwork, where it is heated and passes from below into the burners.

To promote the draught in the digester, the small pipe *S* conducts heated gas into the space below the grate *R*.



The operation of rendering is conducted as follows:—

After filling the jacket *B* with water to the highest point of the flues, and heating the boiler, the digester *A* is charged with the fat to be rendered, care being taken to place the strainer *M* at its highest point. The Argand furnace is heated at the same time by kindling a fire upon the grate *R*.

As soon as the manometer indicates a moderate pressure, the cock on the discharge-pipe *J* is opened and the gases escape to the combustion furnace, where in the mean time sufficient heat must be developed to assure complete combustion of the noisome vapors.

The heating of the apparatus is so regulated that the tension of the steam in the jacket does not exceed four atmospheres and the pressure in the digester is not over 2.5 atmospheres.

To recognize the moment of complete melting, samples are from time to time taken through the pipe *U*.

For the manufacture of margarin, the selected tallow is freed from adhering particles of flesh and blood by first soaking in warm water and afterwards thoroughly washing in cold water. It is next introduced into a hashing machine, and when thoroughly disintegrated melted by steam at  $140^{\circ}$  to  $151^{\circ}$  F. The melted fat is then allowed to stand until it deposits the floating fragments of membrane, which collect on the bottom, forming "scrap." The fat is then crystallized at  $95^{\circ}$  F. and subjected to pressure at this temperature. The residue is "prime press tallow," and is used in the manufacture of candles. The fat pressed out, known as oleomargarin, is used in the manufacture of artificial butter.

By subjecting tallow to pressure at a low temperature, *tallow oil* is obtained, which remains liquid at an ordinary temperature.

Commercial tallow frequently has a dirty gray color. Such tallow, to be worked into stearin by saponification, needs refining, as otherwise candles manufactured from it will not show the white appearance so highly valued. The refining consists in remelting the tallow with water, generally with an addition of common salt, alum, or soda.

A very simple method to bleach tallow is to melt it and then stir in 6 to 10 pounds of soda lye of  $20^{\circ}$  to  $24^{\circ}$  B. for each 100

pounds of tallow and allow it to stand and settle. The brown sediment can be used for rosin grain-soaps.

In a pure state the tallows are nearly inodorous; on exposure to the air for some time, mutton tallow, however, acquires a peculiar odor, which, according to Chevreul, is due to the evolution of a volatile fatty acid which he terms *hircic acid*, the existence of which is, however, doubtful. Tallow consists of stearin, palmitin, and olein, their proportions varying in the fat from the different parts of the animal.

There is no great difference in the saponification equivalents of the various varieties of tallow, which is readily explained by the fact that oleic acid and stearic acid fix nearly the same quantities of alkali, 1 gramme of oleic acid requiring 198.7 milligrammes of caustic alkali for saponification, 1 gramme of palmitic acid 218.9 milligrammes, and 1 gramme of stearic acid 197.3 milligrammes.

The iodine degree of tallow is 40, according to Hübl, and that of the fatty acids separated from tallow, 25.9 to 32.8, according to Demski and Morawski.

Tallow, as found in commerce, is frequently contaminated with water, dirt, particles of skin, etc. Such contaminations are readily detected by the method given on page 102. It is further adulterated with cheaper fats, such as bone fat, kitchen fat, residue from the manufacture of margarin, etc. Such adulterations cannot always be detected with certainty. The best guide is the determination of the melting points, or, still better, that of the fatty acids separated from the tallow, which, according to Dalican, should never be below 111° F.

Tallow adulterated with distilled wool fat has recently made its appearance in commerce. This adulteration can, however, be readily detected by the considerable percentage of cholesterin in the wool fat. Saponify the suspected tallow with caustic potash and shake the resulting soap with ether. The latter absorbs the cholesterin and leaves it behind on evaporation. By adding hydrochloric acid and chloride of iron to the residue a violet coloration is produced.

Adulteration with cotton-seed stearin is detected by melting the fat, crystallizing it in a drying box at 95° F., after eighteen

hours pressing it through a cloth and determining from the filtrate the iodine degree, which, with filtrate from tallow containing cotton-seed oil, is from 75 to 80, and with that from pure tallow about 55.

Before the introduction of palm-oil, cocoanut-oil and palm-kernel oil, tallow was the most important raw material used in the manufacture of soap. It saponifies only with weak lyes, requiring, according to its age, in the commencement of the operation a lye of  $8^{\circ}$  to  $10^{\circ}$  B.; with this it readily forms an emulsion, which, on reaching the boiling point, is converted into a chemical combination. By continuing the boiling with this lye a viscid, thick soap-paste is obtained, which is not the case with any other fat. Generally the boiling is, however, continued with lye of  $12^{\circ}$  to  $15^{\circ}$  B., until the combination forms a clear, viscid paste, the exact fitting of which is recognized by the quick appearance of a gray edge on a sample upon the spatula.

After the introduction of the combination with lye, most fats take up the further lye required very rapidly and well; with tallow this process, however, takes place very gradually, and it is, therefore, necessary to add the lye in portions. By adding the portions of lye in rapid succession, it may happen that the soap-paste, notwithstanding it shows sharpness, is turbid and not saturated. This defect is best remedied by the addition of some weak lye with a moderate fire; the paste gradually clarifies until all sharpness disappears, and only when this is the case will it be possible to determine how much lye is required for complete saponification. It may further occur that by adding too concentrated lye (of about  $20^{\circ}$  B. or more) the combination formed may be entirely destroyed so that lye runs off. This fault is remedied with weak lye or water and boiling slowly. In very difficult cases it is best to interrupt boiling and squirt water over the soap-paste.

Saponification is more readily and better effected by direct steam than by an open fire, since a large quantity of tallow can in a short time be converted into a clear paste free from scum, which, when properly "salted out," also yields a finished grain free from scum, so that for most purposes clear boiling is not required.

100 pounds of tallow saponified with soda lye yield at the



utmost 165 pounds of ground grain-soap, which, however, dries out strongly, so that the bars warp tray-shaped. However, by boiling down solid, as is especially required in boiling over an open fire, 100 pounds give only 150 pounds of soap. By allowing the latter to cool in large frames it shows formation of grain.

*Lard.*—In the United States hog's lard is largely used in the manufacture of soap and candles. It is rendered or melted in a similar manner as tallow; chiefly over an open fire, though in large operations steam is found to present the most economical method. Lard has a granular, salve-like consistency, a pure white color, and agreeable taste; on exposure to air it soon turns yellow and rancid. It consists of 62 parts of olein and 38 parts of solid fat (mostly palmitin), and contains, according to Allen and Thompson, 0.25 per cent. of non-saponifiable substance. By saponification 100 parts of lard yield, according to Braconnot, 8.8 parts of glycerin and 95 parts of fatty acids, which commence to congeal at  $131^{\circ}$  F., and become entirely solid at  $125.5^{\circ}$  F., but according to Mayer their melting point is at  $95^{\circ}$  F., and their congealing point at  $93^{\circ}$  F. The statements about the melting point of lard vary from  $79^{\circ}$  F. to  $107.5^{\circ}$  F. These variations may be largely due to the varying solidity of the fat from different parts of the body and to different methods employed in the determination. The specific gravity of lard is 0.938 at  $50^{\circ}$  F. Its saponification equivalent is, according to Valenta, 195.3 to 196.6, and its iodine degree, according to Hübl, 57.6 to 60.

By subjecting lard to strong pressure at  $32^{\circ}$  F., it is separated into 62 parts of a colorless oil, the so-called "lard-oil," and 38 parts of a solid mass, consisting of tallow (margarin), palmitin, and stearin. Lard-oil has a pale color and mild taste. It is used in the fabrication of soaps and pomades, as a lubricant, and in the wool industry. It has a specific gravity of 0.915 at  $50^{\circ}$  F., and begins to separate stearin at below  $32^{\circ}$  F. Its saponification equivalent is, according to Moore, 191 to 196. The solid portion is brought into commerce under the name of "solar stearin," and furnishes an excellent material for the manufacture of candles.

Lard is frequently adulterated, the principal sophistication being with water; alum or lime is sometimes added to enable it to absorb large quantities of water. An addition of 1 per cent.

of slaked lime or of 2 to 3 per cent. of alum will cover 10 to 12 per cent. of water. By melting and recondensing the lard, the foreign substances form a deposit on the bottom and can be readily determined. Some tallow is generally added to give the lard a firmer appearance.

The behavior of lard towards lye is similar to that of tallow; the older and more rancid it is the quicker saponification is accomplished, while the fresh neutral fat requires in the commencement a weak lye. Lard is much liked for the preparation of smooth white grain-soaps. Though lard by itself gives a very white soap, even if by bad treatment it has acquired a yellowish or dirty gray color, it is, on account of the lardaceous and somewhat soft consistency of the soap, better adapted for working in connection with palm-kernel oil and cocoanut-oil, which give lean and brittle soaps, an addition of one-third or two-fifths of lard being sufficient in order to obtain a beautiful, solid, and delicate soap. Good lard is at present used only for toilet soaps.

The lard from animals that have died by sickness has generally a dark color and bad odor. In connection with palm-kernel oil it yields good results; the best proportions are 2 parts of lard to 3 of palm-kernel oil or 3 parts of lard to 5 of oil. A larger quantity of lard might yield a soft or mottled product.

The yield of pure ground grain-soap will generally reach about 155 per cent., though the softer a fat the lower the yield of pure grain-soap.

*Horse fat.*—Generally speaking, the horse is poor in fat. The careless manner in which it is prepared from the carcass renders it rather repulsive, yet, when extracted from the recently slaughtered animal, it constitutes a very suitable and good material for the manufacture of soap. Horse fat varies in consistency according to the organs from which it is taken and the care given to its production. It is either solid, forming a real tallow, or is more or less of the consistency of lard. The fat found in commerce has generally a more or less dark color, a bad odor, and the consistency of lard. As a rule it is not pure horse fat, but contains lard, bone fat, etc. By treating with strong lye in a similar manner as described for lard, it can be bleached, and forms then a very good material for white soaps, while the dark sediment

can be utilized for dark soaps. Most of the fat being quite rancid, combination is effected with a medium lye, complete saponification taking place with ease and rapidity. The application of this fat for household soaps is about the same as that of lard. Its peculiar sweetish odor makes it a suitable addition to palm-kernel oil to weaken the strong odor of the soap prepared from the latter.

*Bone fat.*—The bones of all animals contain up to 3 per cent. of fat. Though it has not been thoroughly studied, the constitution of bone fat seems to be about the same as that of other animal fats, it being only richer in the glyceride of oleic acid, and, therefore, softer and easier to melt. To produce the fat the bones are broken as much as possible in a lengthwise direction. They are then brought into a kettle partly filled with water and heated to the boiling point, which causes the fat to float on the surface, where it is skimmed off with a flat iron spoon and passed through a sieve, which retains the solid particles. When it is noticed that no more fat separates, the bones are taken out by means of a large perforated shovel and replaced by fresh ones, so that the water may be used several times.

Most bone fat is now obtained as a by-product in the manufacture of animal charcoal or bone meal. The bones, before their mechanical comminution, are either steamed in closed iron cylinders for a few hours at a pressure of 2 to 4 atmospheres, or extracted in a special apparatus with benzene. By the latter method the bones are more completely exhausted than by steaming, but the extracted fat has a strong odor of benzene which is difficult to remove. It is purified by remelting upon salt water and introducing steam for some time.

The fat obtained by boiling fresh bones has a white to yellowish color, a slight odor and taste, and is of a smeary consistency. It consists of stearin, palmitin, and olein. When thoroughly purified it keeps well and makes a good lubricant. The bone fat obtained as a by-product in the manufacture of bone meal is largely obtained from old, partly putrefied bones, and is of a dark color and unpleasant odor. It always contains a considerable quantity of free fatty acids and frequently calcium lactate and



calcium sebate, the latter permitting the incorporation of considerable water.

The melting point of commercial bone fat varies from  $68^{\circ}$  to  $83^{\circ}$  F. The fatty acids of the fat from fresh bones melt, according to Hübl, at  $86^{\circ}$  F. and congeal at  $82.5^{\circ}$  F. Valenta determined the saponification equivalent at 190.9, and Demski and Morawski the iodine degree of the separated fatty acids at 55.7 to 57.3. In examining bone fat, attention must be especially directed towards dirt and water.

The ordinary bone fat of commerce is difficult to bleach ; the frequently recommended methods with potassium bichromate and sulphuric acid or hydrochloric acid accomplishing the object in but few cases.

The commercial bone fats contain a large quantity of free fatty acids, and, therefore, combine readily with strong lyes containing a considerable percentage of alkaline carbonates. In other respects their constitution varies, however, very much. There are, for instance, bone fats which in color and consistency resemble a poor quality of tallow and furnish a very good material for the manufacture of soap, while others resemble poor distilled oleic acid, and are incapable of forming cohering grain-flakes when boiled by themselves. Even with the better qualities of bone fat, the sub-lye is generally more or less turbid, and, on cooling, forms a gluey skin in consequence of the impurities contained in the fat. The yield varies very much. Good, solid bone fat may yield as much as 150 to 155 per cent. of good ground grain-soap, which is quite solid and lardaceous, though not so white as tallow grain-soap.

Bone fat by itself is but little employed in the fabrication of soap, but much in connection with other fats. It is chiefly used for resin grain soaps, it being less adapted for smooth white grain soaps, as even the best bone fat does not give the pure white product so desirable for this variety. It can also be used in connection with other fats for soft soaps, the lighter varieties being well adapted for grained soaps, however, only when the appearance of the product is of secondary importance, as is the case with soaps for the textile industry. For oil soaps only a small quantity can be worked in summer in connection with linseed-oil.

*Wool fat.*—The wool of sheep contains a considerable quantity of a fatty body consisting chiefly of cholesterin, isocholesterin, cholesteryl stearate and palmitate, and other compound ethers of cholesterin, and can, therefore, be but incompletely saponified. Moreover, the so-called wool sweat (suint) contains a considerable quantity of potash soaps. The fat extracted from the crude wool by means of ether or bisulphide of carbon has a yellowish color and the peculiar odor of wool.

For the manufacture of soap wool fat is of but little value; it cannot be worked by itself, and is chiefly used for resin soaps.

Some years ago a method for purifying wool fat and mixing the purified article with water was patented. It forms a white salve-like mass, to which the term "lanolin" has been applied, and is used as a vehicle for salves and pomades.

Wool fat can be distilled with superheated steam, and then forms a white or yellowish mass, which melts at about  $107.5^{\circ}$  F., and recondenses at  $104^{\circ}$  F. It is frequently used as an adulterant of tallow (see tallow).

*Glue fat.*—In making glue from hides, tendons, etc., much fat is collected, which, if well prepared, can be usefully employed in making soap. The commercial article contains considerable lime and other impurities, which can, however, be removed with dilute sulphuric acid. When the fat is boiled in a five per cent. solution of sulphuric acid for about one hour, the lime and other impurities are carried down with the water, the clear grease floating on the surface, whence it is ladled off. This fatty acid will make with soda lye, with or without resin, a good and firm soap, useful for all domestic purposes. It should be boiled, for in the cold soaps it would not answer so well.

*Neat's-foot oil.*—This fat is obtained from the bones of the legs and feet of cattle and sheep. If this product were abundant, it could be usefully applied in making soaps of good quality, but it is generally used as a lubricant and for dressing leather for which it is admirably adapted. It is of a greenish-yellow color, and when fresh has no odor, is limpid at ordinary temperatures, becoming solid in the cold. Its specific gravity is 0.915 to 0.916 at  $59^{\circ}$  F. It forms with soda lye a very fine white soap, partak-

ing of the nature of the fat, being somewhat soft, oleic acid being the largest constituent of the oil.

*Fish-oils and train-oils.*—In this category are classed the oils derived from various marine animals, which in commerce are generally designated by the names of their sources, as whale, seal, cod-liver oil, etc. They are fluid at an ordinary temperature, and have a peculiar disagreeable odor and taste, due to the admixture of volatile fatty acids. In these oils, the oleic acid occurring in other oils, is partly replaced by physetoleic acid. The oils derived from several marine mammalia contain combinations which are not glycerides, but ethers of the higher fatty alcohols.

All train-oils are sparingly soluble in cold alcohol, somewhat more freely in hot alcohol, and very readily in ether. Most of them are blackened by gaseous chlorine.

Train-oils are chiefly adulterated by compounding a good quality with a poorer one. Such sophistication can scarcely be detected, as it is difficult to distinguish the separate oils, their specific gravity varying but little, being between 0.915 and 0.930. Neither do the melting points of the separated fatty acids, the saponification equivalents and iodine degrees furnish a sufficient guide for the examination of train-oils.

An admixture of foreign fats is recognized by mixing 1 part of train-oil with 2 parts of concentrated sulphuric acid in a tall beaker glass. If the train-oil is free from foreign fat, a clear mixture is obtained.

Various means are applied in purifying the bad smelling and dark-colored train-oils. Shaking with milk of lime, with diluted potash or soda lye, common salt, and copperas, is a common device, as well as filtering with wood ashes. According to Davidson, train-oil should be shaken with a decoction made of oak bark, then have mixed with it 4 parts of chloride of lime (bleaching powder) stirred into 12 parts of water, permitting it to clear off, when a thick whitish mass will be separated, to which is added dilute sulphuric acid to settle the lime which becomes free. This is an excellent means for disinfecting train-oil, since by this treatment it loses the greater part of its disagreeable smell, so that such train-oil before saponification appears almost odorless. But, nevertheless, the smell reappears when the oil is



converted into soap. Hence train-oil can only be used in manufacturing very common soaps, or by mixing small quantities of it with other fats.

The head cavity of the sperm whale, called by the whalers the case, contains an oily fluid, which, after death, concretes into a granulated substance of a yellowish color, called spermaceti. It is separated from the fluid portion (spermaceti-oil) by filtering, pressing, boiling with some potash or soda lye, and rinsing in cold water. Spermaceti is used, especially in England, in the manufacture of fine candles, while the spermaceti-oil forms an excellent lubricant.

#### FATS AND OILS OF VEGETABLE ORIGIN.

The vegetable fats and oils applied to the manufacture of soap are very numerous and valuable and are found in the fruits, seeds, etc., of plants. The most important solid fats are cocoanut-oil, palm-oil, and palm-kernel oil. There are various other solid fats found in tropical countries, such as shea butter, illipe-oil or bassia-oil, piney tallow, Chinese tallow, cocoa butter, dika-oil, butter of nutmeg, etc., which are very suitable for the manufacture of soap, but are little used on account of their high price, and will therefore be only briefly mentioned. Of the fluid vegetable fats are used: olive-oil, sesame-oil, peanut-oil, castor-oil, cotton-seed oil, linseed-oil, hemp-oil, niger-oil, cameline or German sesame-oil, and small quantities of colza-oil and poppy-seed oil.

*Cocoanut-oil.*—This valuable oil is obtained by boiling or pressing the ground or crushed kernels of the nuts of *Cocos nucifera*, the cocoanut palm, which is found in nearly all tropical countries. Most of the oil is imported from Ceylon and Cochin China, though recently the dried kernels, called “coprah,” are exported to Europe, where the oil is extracted either by expression or extraction. Coprah contains 60 to 70 per cent. of fat. Two varieties are known in commerce, sun-dried and kiln-dried coprah.

Fresh cocoanut-oil has a beautiful white color, a mild taste, and a peculiar, though not disagreeable, odor. It becomes soon rancid

and acquires a somewhat disagreeable, acrid taste and odor. Entirely fresh oil melts at  $68^{\circ}$  F.; the commercial oil from Ceylon and Cochin China shows a melting point of about  $75^{\circ}$  F., and the Brazilian cocoanut-oil (from *Elaeis butyracea*) melts at about  $80.5^{\circ}$  F. By subjecting the coprah to a moderate cold pressure an oil is obtained which melts below  $68^{\circ}$  F. and congeals at  $53.5^{\circ}$  to  $55.5^{\circ}$  F., becoming heated thereby to  $59^{\circ}$  F.

Cocoanut-oil contains chiefly the glycerides of laurostearic, myristic, palmitic, and caprylic acids, and small quantities of capronic and capric acids. St. Evre and Bromeis claimed to have found special fatty acids, which they termed cocinic acid; but Heintz has shown that St. Evre's cocinic acid is a mixture of laurostearic acid with capric acid, and Bromeis's cocinic acid a mixture of laurostearic acid with myristic acid.

Cocoanut-oil has the highest saponification equivalent of all the fats hitherto examined, by which it can be readily distinguished from all other fats, with the exception of palm-kernel oil, which comes next in this respect. Valenta examined several cocoanut-oils and found their saponification equivalent to be from 257.3 to 268.4. The iodine degree of cocoanut-oil is, according to Hübl, 8.9, and that of the separated fatty acids, according to Demski and Morawski, 8.39 to 8.79. The separated fatty acids melt at  $76^{\circ}$  to  $77^{\circ}$  F. and congeal at  $66^{\circ}$  to  $69^{\circ}$  F.

On account of its large percentage of laurostearin, the action of cocoanut-oil in the process of saponification is quite different from that of tallow and most other fats. It requires strong caustic lyes and forms soaps which can only be separated by concentrated solutions of common salt, and then become so extraordinarily hard that they cannot be cut. For this reason a clear boiling to the solid would in case of cocoanut-oil soap be entirely contrary to the end in view and very difficult. While, furthermore, tallow, for instance, treated with very strong lye, floats on top and then can scarcely or not at all be saponified, in the case of cocoanut-oil just the contrary happens. It does not form that milk-like mixture (emulsion) with weak lyes by which the process of saponification is usually preceded, but floats as a clear fat above; only when, by continued boiling and evaporation, the lye has reached a certain strength, the saponification suddenly ensues. For sapon-

ifying cocoanut-oil lyes of such strength are used that the soap, with the lye, receives the intended contents of water, and a separation becomes, therefore, unnecessary. Of course, the amount of the alkali must be so accurately calculated that the soap receives no excess of alkali, or at least but very little.

Cocoanut-oil can also be saponified in the so-called cold way, *i. e.*, can be converted into soap by stirring in strong caustic lyes at a temperature little above the melting point of the oil. Soaps from cocoanut-oil have the property of absorbing a great deal of water without suffering in firmness and appearance. They dissolve freely in water, yielding a strong lather, which is, however, not so durable as that from tallow soap. Soaps from pure cocoanut-oil have the disagreeable property, even if they do not contain an excess of alkali, of producing a burning sensation and redness upon a sensitive skin, and being strongly inclined towards rancidity, whereby they acquire a bad odor and unsightly appearance.

Three principal kinds of cocoanut-oil are at present known in commerce—Cochin China-oil, Ceylon-oil, and coprah-oil—the first being much the best and purest in color. It is best adapted for cold saponification, but is only good when not too old. When old oil of a certain degree of rancidity is stirred together with strong lye, the mass becomes thick too quickly and the soap shows formation of granulation. Commercial Ceylon-oil is generally quite rancid, and, therefore, not well adapted for cold saponification, and besides, the soaps prepared from it are not pure white, but of a grayish shade. Coprah-oil is not very rancid, and, therefore, suitable for cold saponification; it does not yield, however, pure white soaps, and hence is not adapted for the fabrication of toilet-soaps. This defect can, however, be remedied by the following method of refining: Boil 750 pounds of coprah-oil with 15 pounds of soda lye of 6° B. and 10 pounds of water for half an hour, constantly removing the scum. Then add 1½ pounds of common salt, remove the scum, and let the whole boil half an hour longer. Repeat the boiling with 1½ pounds more of common salt and let the oil stand over night for the salt water to settle.



*Palm-oil*.—Vast quantities of this oil are consumed in the fabrication of soap, especially in England, where it was first used. It is obtained by boiling or pressing the fruits of various species of palms, chiefly of *Avoira Elaeis* or *Elaeis guianensis* and *Elaeis melanococca*. The principal places of production are Western Africa (Guinea) and South America (Guiana), while some of it is brought into commerce from the Canaries, Madeira, and other places. The largest quantity and best quality of palm-oil come from the West African coast south of Sinoe, in the Republic of Liberia, to Cameroon, in the Bay of Benin. The fruits of *Avoira Elaeis* are dark orange-yellow, almost brown, of the size of a pigeon's egg, and contain a solid kernel under a fleshy cover. The latter yields the product known as palm-oil, which is directly brought into commerce from the localities where the palms grow. The kernels also yield an excellent oil, known as palm-kernel oil, the manufacture of which is, however, principally carried on in Europe, where it is either expressed or extracted by means of carbon bisulphide or benzene.

Palm-oil is of an orange-yellow to red-brown color, of a butyraceous consistency, and, when not rancid, has the odor of orris-root. The melting point of fresh palm-oil is at about  $80.5^{\circ}$  F., while that of rancid oil is much higher, rising to  $108.5^{\circ}$  F. The melting point of the fatty acids separated from palm-oil varies between  $116.5^{\circ}$  and  $118.5^{\circ}$  F., and their congealing point between  $104^{\circ}$  and  $113^{\circ}$  F. The saponification equivalent of palm-oil is, according to Valenta, 202 to 202.5, and that of the separated fatty acids 206.5 to 207.3. The iodine degree is, according to Hübl, 51.5.

Palm-oil consists chiefly of palmitin and olein. A very characteristic feature of this fat is the great quantity of free fatty acids it contains. In fresh palm-oil has been found one-third and in an old sample as much as four-fifths of its weight of free fatty acids. The greater portion of glycerin separates as such and can be obtained by extraction with water.

The reddish-yellow color of palm-oil is not destroyed by saponification, so that the soap manufactured from the crude product shows a yellow color. In the manufacture of stearin the coloring

substance can be destroyed by acid saponification and distillation, but not by ordinary lime saponification.

If white soaps are to be manufactured, the palm-oil must first be bleached. This is done either by heating to a certain degree, by heat and air, or by chemical agents.

*The application of heat, i. e.*, heating the oil to from  $428^{\circ}$  to  $464^{\circ}$  F., is the simplest method of bleaching. Impure oil must, however, first be purified by melting upon water at a moderate heat. The supernatant clear fluid is then drawn off from the sediment, brought into an iron kettle, and quickly heated to  $396^{\circ}$  F., which suffices for most varieties of palm-oil, though some require a higher temperature. For the removal of the noxious vapors the kettle is covered with a well-fitting lid, in which an iron pipe is inserted, which opens into the chimney of the fire-place.

Klepzig,\* who is very much in favor of bleaching by the application of heat, quickly brings the temperature to  $392^{\circ}$  F., and then heats carefully to from  $419^{\circ}$  to  $428^{\circ}$  F., keeping up the latter temperature for one hour without stirring. In about half an hour the oil has acquired a lemon color and is perfectly clear, the yellow coloration disappearing, as a rule, entirely in  $1\frac{1}{2}$  hours, and the oil becoming dirty gray. By pouring a few drops of the oil upon a plate, fine particles of carbon will be observed floating in them. The fire is then withdrawn and the oil allowed to stand quietly in the kettle. With some experience it can be readily judged whether the oil is to be kept for some time longer at the indicated temperature or heated to a still higher degree.

Oil purified before heating shows, when cold, a whitish color shading into brown; oil not previously purified is dirty gray from the many finely-divided particles of carbon suspended in it. These impurities, however, do not injure the oil for the manufacture of soap, as they are salted out. Klepzig was of the opinion that bleaching was effected by this finely-divided carbon; this, however, is not correct, since well-purified oils, in which the formation of carbon is very slight, are also bleached by the application of heat. This method of bleaching simply depends

\* Die Palmölbleiche durch Kohlenstoff. Leipzig, 1857.

upon the fact that the coloring substance of palm-oil is destroyed by heat. It has the defect that the bottoms of the kettles suffer much. With careful work and good, pure palm-oil the loss in weight is 1 to  $1\frac{1}{2}$  per cent.

To bleach palm-oil by the *agency of heat and air*, it is heated to  $212^{\circ}$  F., and at this temperature brought in contact with the air which is accomplished by various mechanical appliances. The most simple is to fill a fine-meshed sieve fastened to a long handle with oil, raise it up quickly, and bring the oil in contact with the atmospheric air by allowing it to run back into the kettle. By frequently repeating this rather slow process the oil is bleached, though as a rule incompletely. A better arrangement is a sort of paddle wheel so adjusted that one-half of it dips into the oil. By setting the wheel in motion the paddles lift up a portion of the oil, allowing it to run back in a fine stream into the kettle. Though this mode of bleaching has the advantage of not injuring the agreeable odor of the oil it is now seldom practised.

*Bleaching by chemical agents* is effected by oxidizing substances, the best being chromic acid, *i. e.*, a mixture of potassium bichromate and hydrochloric acid. The process as executed in most soap factories is as follows: The palm-oil is first purified by melting upon water. After allowing the impurities to settle the supernatant clear oil is drawn off and allowed to cool to  $122^{\circ}$  F. To 1000 pounds of oil are then added with constant stirring 50 pounds of hydrochloric acid and 12 pounds of potassium bichromate previously dissolved in 24 pounds of boiling water. After stirring ten to fifteen minutes the oil shows a dark, dirty gray color. Sometimes a few pounds of sulphuric acid are added. The stirring is continued until the oil is entirely clear with a bluish lustre. 60 to 80 pounds of hot water are then poured over the oil by means of a watering-pot, and after covering the vat it is allowed to stand till the next day.

With most varieties a much smaller quantity of potassium bichromate suffices, 1000 pounds of oil frequently requiring only 5 to 6 pounds. In a soap factory which we had occasion to visit, where much palm-oil is bleached, the process is carried on according to the method recommended by Bolley. 1000 pounds of melted palm-oil are brought at  $122^{\circ}$  F. into a wooden vat; 5 pounds of



potassium bichromate are then added, and, after thorough stirring, 10 pounds of hydrochloric acid, and finally  $\frac{1}{2}$  pound of sulphuric acid. Stirring is then continued half an hour and a sample taken. If the oil proves still yellow, some more potassium bichromate, hydrochloric acid, and sulphuric acid are added until the desired result is obtained. The oil is then brought into a copper kettle and boiled for some time with water to remove all constituents of the bleaching agent. It is then allowed to stand covered for some time, and finally the supernatant clear fat drawn off.

Palm-oil bleached by means of potassium bichromate has frequently a greenish shade, which is due to a small quantity of chromic oxide contained in it. This can be extracted, and a faultlessly white product obtained by boiling with dilute hydrochloric acid and remelting the oil upon pure water.

Commercial palm-oil varies very much in quality, the *prima lagos* and *secunda lagos* being the best, the former being more readily bleached than the latter. Next in quality is the *old Calabar-oil*, which is also readily bleached, especially by chemical means. The oils from *Accra* and *Benin* are not so pure as the preceding ones, and very unequal in their behavior during bleaching. They are generally used for dark grain-soaps and rosin grain-soaps. The crude oils from *Cameroon*, *Gaboon*, and *Liberia* are generally remelted on board the vessels and brought into commerce under the name of *purified Cameroons*. They are generally very rancid, and, notwithstanding their name, by no means free from dirt. They cannot be as readily bleached as lagos and old Calabar-oil, and generally require chemical agents for the purpose.

How much commercial palm-oils vary is shown by the following table published by H. Yssel de Schepper and A. Geitel,\* which gives the percentage of water, dirt, and neutral fat of a large number of palm-oils, and the congealing points of the fatty acids obtained from them:—

\* Dingler's Polytechn. Jour., 245, 295.

Name.	Water. Per cent.	Dirt. Per cent.	Neutral fat. Per cent.	Congeaing point of the fatty acid. Degree F.
Congo . . . . .	0.78-0.95	0.35-0.7	16-23	114.5°
Saltpont . . . . .	3.5-12.5	0.9-1.7	15-25	79
Addah . . . . .	4.21	0.35	18	111.3
Appam . . . . .	3.60	0.596	25	114
Winnehah . . . . .	6.73	1.375	20	114
Fernando-Po . . . . .	2.68	0.85	28	114.5
Brass . . . . .	3.05	2.00	35.5	113
New Calabar . . . . .	3.82	0.86	40	113
Niger . . . . .	3.0	0.70	40-47	113
Accra . . . . .	2.2-5.3	0.60	53-76	111.2
Benin . . . . .	2.03	0.20	59-74	113
Bonny . . . . .	3.0-6.5	1.2-3.1	44-88.5	112
Great Brassa . . . . .	2.4-13.1	0.6-3.0	41-70	112
Cameroons . . . . .	1.8-2.5	0.2-0.7	67-83	112
Cape Labon . . . . .	3.6-6.5	0.7-1.5	55-69	105.8
Cape Palmas . . . . .	9.7	2.70	67	107.7
Half Jack Jack . . . . .	1.9-4.2	0.7-1.24	55-77	102.2-106.3
Lagos . . . . .	0.5-1.3	0.3-0.6	58-68	113
Loando . . . . .	1.5-3.0	1.0-1.9	68-76	112
Old Calabar . . . . .	1.3-1.6	0.3-0.8	76-83	112
Gold Coast . . . . .	1.98	0.50	69	105.8
Sherbo . . . . .	2.6-7.0	0.3-1.2	60-74	107.6
Gaboon . . . . .	2.0-2.8	0.3-0.7	70-93	112

To obtain the oil from the sediment formed by storing large quantities of palm-oil, the sediment is boiled upon water, and after standing for some time the supernatant clear oil drawn off. The slimy residue is brought into large barrels, and after adding, with constant stirring, some sulphuric acid, and continuing the stirring for some time, the barrels are covered and the contents allowed to settle. After a few hours the dark-colored oil, which has separated, is drawn off, and can be either bleached with potassium bichromate and acid or immediately used for dark soaps.

The sediment obtained by bleaching with chemical agents also contains much oil, and is, therefore, washed with hot water with an addition of sulphuric acid. The oil obtained, though dark, yields quite pale soaps, which in odor and firmness are not inferior to other palm soaps.

Palm-oil was formerly much adulterated, and substitutes for it consisting of mixtures of wax, tallow, and lard colored with turmeric and scented with orris root, were even found in commerce. Such adulterations and mixtures could be readily detected

by the test with glacial acetic acid, which dissolves palm-oil, but leaves all the rest undissolved. The addition of turmeric is recognized by the brown color formed on stirring in soda lye. With the present prices of palm-oil such adulterations need, however, not be feared, as they would not pay.

All palm-oils, no matter how much they may vary in quality, yield a firm soap of an agreeable odor, the scent remaining quite perceptible in combination with other fats and oils and even with resin.

Palm-oil, crude or bleached, is readily saponified. It yields, even with weak lye of  $8^{\circ}$  B., a quite thick and viscid soap paste. Generally it is, however, saponified with lye of  $12^{\circ}$  to  $15^{\circ}$  B., yielding, when thoroughly salted out, an abundant grain quite free from scum, which, if the paste was entirely clear, is thoroughly saturated. Since palm-oil is generally quite rancid, and contains, therefore, free fatty acids, a lye of  $15^{\circ}$  B., which, with a high percentage of lime, always contains some alkaline carbonate, is completely exhausted, especially when adding it carefully towards the end of the operation and boiling slowly. Saponification with direct steam is especially quick and perfect, the grain separating almost entirely clear, the best proof of a complete saponification.

On account of the large content of palmitic acid, palm-oil yields a good firm soap, notwithstanding the large percentage of water retained even by grain-soap. Palm-soap, when made from pure oil with soda lye, will become with age too hard to lather well. This defect can be overcome in two ways, either by using about 2 per cent. of potash lye with the soda lye, or by adding about that quantity of resin to the oil used, the latter making a soap that from its solubility and free lathering is quite popular with most people.

In a variety of toilet-soaps palm-oil enters to great advantage, for if the oil be properly refined and bleached the odor is exceedingly pleasant and combines very well with nearly all the volatile oils commonly used in soaps; besides, the soap will require a less quantity of the expensive oils or perfuming substances. One hundred pounds of pure oil yield 162 to 165 pounds of fresh ground soap.



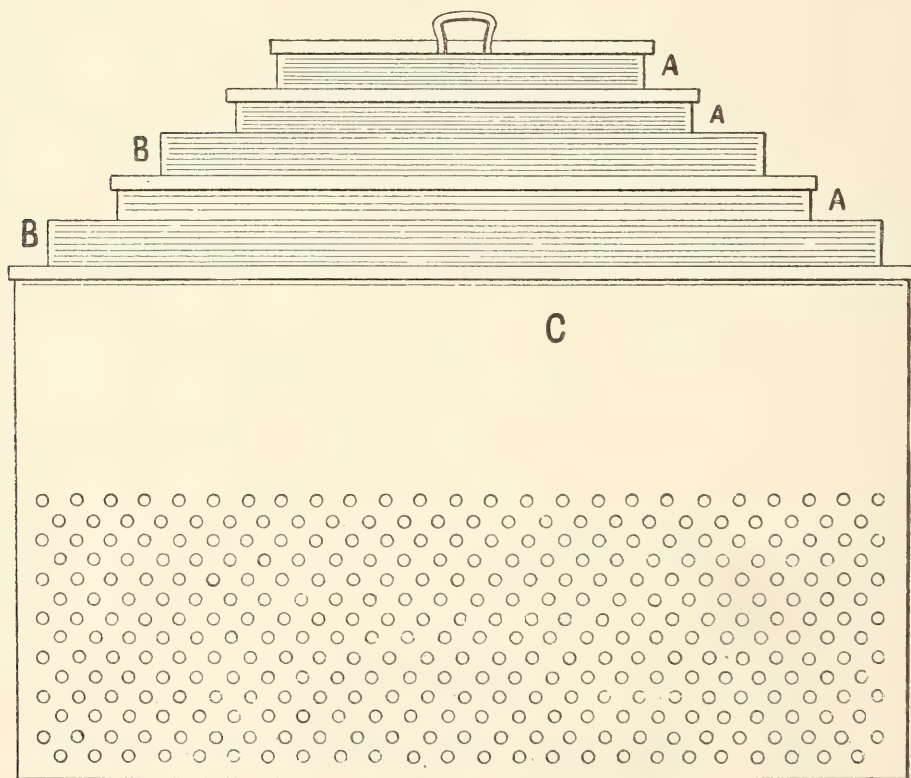
The principal use of palm-oil, besides the fabrication of soap, is in the manufacture of stearin.

*Palm-kernel oil.*—The kernels of the palm fruit were formerly thrown away, but, as previously mentioned, are now brought to Europe, where the oil is obtained either by expression or extraction with carbon bisulphide or benzene. The oil obtained by expression is yellower than that by extraction.

The kernels reach the market freed from the woody shell. The percentage of oil varies between 35 and 50 per cent., though some kernels, it is claimed, contain as much as 60 per cent. and more.

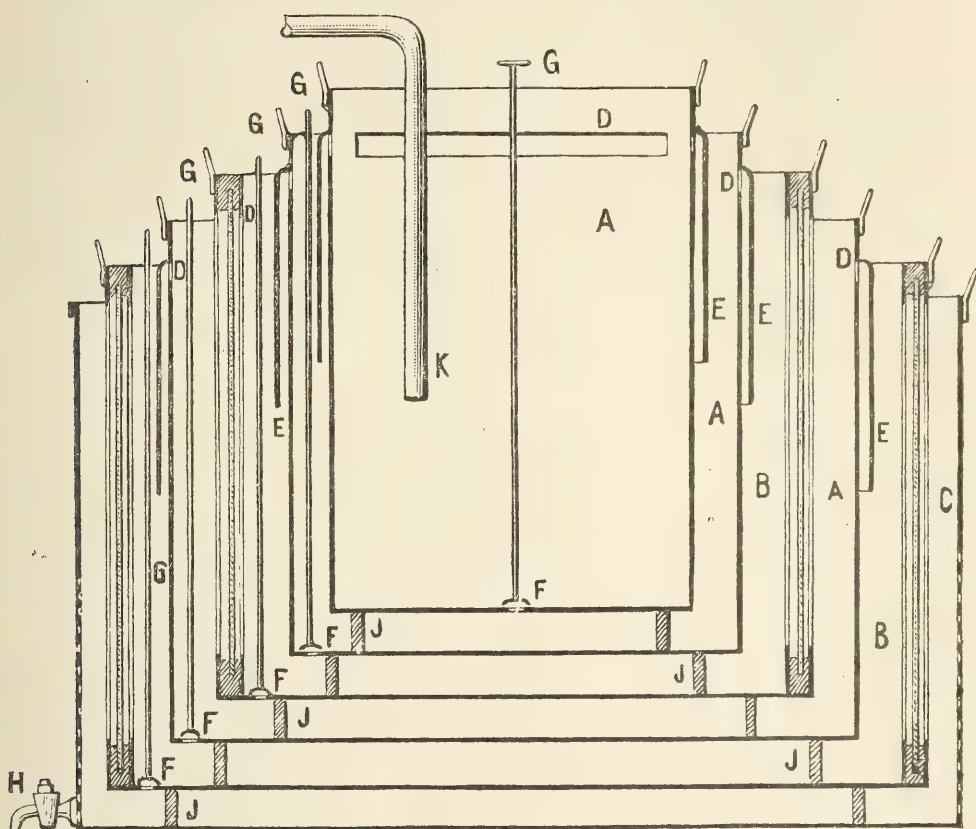
As the oil obtained by expression contains vegetable albumen and mucus, it must be allowed to rest for a long time in a melted state for purification and clarification. Purification by sulphuric

Fig. 19.



*A*, decanting boxes ; *B*, filtering boxes ; *C*, sieve-box ; *D*, slit in *A* ; *E*, jacket around *A* ; *F*, discharge-valves ; *G*, valve-rods ; *H*, discharge-cock ; *J*, supports ; *K*, feed-pipe.

Fig. 20.



acid being tedious, Schneider's apparatus, shown in Figs. 19 and 20, is well adapted for the purpose.

The apparatus is very compact, but can be easily taken apart and cleansed. It contains three series of boxes made of wood and sheet-iron, viz., the decanting boxes *A* and the filtering boxes *B*, which sit in the sieve-box *C*. All the boxes are open on the top. The boxes *A* consist each of a bottom and four vertical sides, each provided with a vertical slit *D* near the upper edge. The jacket *E* surrounds the boxes and joins their sides above the slit *D*. The filtering boxes *B* consist of bottoms and framework sides, the latter being covered with linen or some other filtering material. The lower halves of the sides of the sieve-box *C* are perforated.

The filtering boxes *B* and the sieve-box *C* are provided with valves *F*, which are set by the valve-rod *G*; the sieve-box is further provided with a discharge-cock *H*.

The apparatus works in the following manner: The sieve-box *C* is suspended over the reservoir which is to receive the clarified oil, and the decanting boxes *A* and filtering boxes *B* are placed upon the supports *J* in the sieve-box *C*. The oil to be clarified is introduced through the pipe *K* into the innermost decanting box *A*, passes through the slit *D* into the second box *A*, and from here into the filtering box *B*, and after penetrating through the filtering material with which the sides are covered, arrives finally in the sieve-box *C* and leaves it through the perforations.

The impurities are precipitated on the bottoms of the boxes and the filtering material, which, of course, must be occasionally cleansed. This is effected by means of the valves *F* and the cock *H*.

Palm-kernel oil consists chiefly of the glycerides of lauric, stearic, palmitic, and oleic acids, and contains only small quantities of tricaprin, tricaprylin, and tricapronin (and perhaps some trimyristin) in about the following proportions: Triolein 26.6 per cent.; tristearin, tripalmitin (trimyristin?) 33 per cent.; trilaurin, tricaprin, tricaprylin, tricapronin 40.4 per cent.

The melting-point of fresh palm-kernel oil is at from  $77^{\circ}$  to  $79^{\circ}$  F.; that of old rancid oil being somewhat higher. Its saponification equivalent is, according to Valenta, 247.6, and that of the separated fatty acids 265.8. By the determination of the saponification equivalent the fatty acids of palm-oil can be readily distinguished from those of palm-kernel oil. The iodine degree of palm-kernel oil is, according to Hübl, 13.4 to 13.6, and that of the separated fatty acids, according to Demski and Morawski, 12.07.

The behavior of palm-kernel oil, as regards saponification, resembles that of cocoanut-oil; the behavior of both these fats, though not exactly alike, being, no doubt, due to the large percentage of laurostearin characteristic of them. Palm-kernel oil requires strong caustic lyes, though of a less degree than cocoanut-oil. While palm-kernel oil combines quickest with an initial lye of  $26^{\circ}$  to  $30^{\circ}$  B., and the combination takes place with greater difficulty the more these degrees are exceeded, cocoanut-oil readily forms a combination with much stronger lye. In their behavior towards common salt the soaps from the two fats also resemble



each other, but are not alike. The salting out of soaps from palm-kernel oil, though difficult, is effected with greater ease than that of soaps from cocoanut-oil.

It has been frequently asked whether there is a difference between expressed and extracted palm-kernel oil; at the present time this question can be unreservedly answered with "No." Cases, formerly referred to by different writers, of soap from extracted oil turning entirely black in boiling, with the development of an intense odor of carbon bisulphide, are scarcely possible now, and were, no doubt, due to extracting the oil with impure carbon bisulphide and insufficient subsequent purification.

The manner of detecting carbon bisulphide in extracted oil has been given on page 108. The oil can be freed from carbon bisulphide, or rather from its products of decomposition, by boiling it for some time with salt water by the introduction of steam. To test whether the desired result is attained, take from time to time a sample of the oil, filter it through paper until entirely clear and saponify it. The resulting soap must not show a bad odor.

*Shea butter, galam butter or bambuk butter* is obtained by boiling with water the comminuted kernels of *Bassia Parkii*, a tree belonging to the *Sapotææ species*, growing in the interior of Africa. The fat is of a butyraceous consistency, gray-white or greenish-white, of a peculiarly viscid and sticky nature (similar to a mixture of fat and turpentine), and a peculiar, aromatic odor. It keeps a long time without turning rancid. It melts at between  $82.5^{\circ}$  and  $84^{\circ}$  F., though statements of various observers vary very much in this respect. With soda lye it yields a very hard, white soap.

*Mahwa butter, Illoopa oil or Bassia oil* is obtained from the seeds of *Bassia longifolia* and *Bassia latifolia*. The fresh fat is yellow or greenish-yellow, and has an agreeable and mild taste and odor. On exposure to air and light, the coloration disappears quite rapidly and the fat becomes rancid. It dissolves partly in alcohol and completely in ether, carbon bisulphide, benzol, etc. It is readily and completely saponified, yielding a hard, white soap of an agreeable odor, which is capable of fixing a considerable quantity of water without losing firmness.

According to Valenta, mahwa butter melts at  $78^{\circ}$  F. and congeals at  $63.5^{\circ}$  to  $65^{\circ}$  F., while the fatty acids separated from it melt at  $103^{\circ}$  F. and re-congeal at  $100.5^{\circ}$  F. The fat consists of palmitin and olein; its saponification equivalent is 192.3.

*Piney tallow*, also called *vegetable tallow* or *vateria fat*, is obtained from the seeds of *Vateria indica*, L., a tree indigenous to the East Indies. The air-dried seeds contain 49.21 per cent. of a solid, greenish-yellow fat, which soon becomes white on exposure to light, and has a peculiar slightly balsamic odor. The fat prepared from commercial seeds shows a melting-point of  $107.5^{\circ}$  F., while, according to other statements, it melts at  $98^{\circ}$  F. and congeals at  $87^{\circ}$  F. It saponifies with great ease, 1 gramme of fat requiring 191.9 milligrammes of caustic potash, whereby 8.4 per cent. of glycerin is separated. The fatty acids separated from the products of saponification melt at  $134^{\circ}$  F. and congeal at  $130.5^{\circ}$  F., and consist of a mixture of oleic acid and solid fatty acids. The latter amount to about 60 per cent. of the weight of the vegetable tallow and melt at  $147^{\circ}$  F. Notwithstanding this high melting-point, the pre-eminently crystalline product is rather soft and readily friable.

*Chinese tallow* is obtained from the seeds of *Stillingia sebifera*, or the tallow tree indigenous to China. The fruit of the tree is a black, somewhat globular capsule, and contains three egg-shaped seeds coated with a thick, hard, tallowy layer, while the seed-kernel contains a liquid fat. The tallow is obtained by one of two methods. The seeds freed from the capsules are treated with steam, whereby the tallow (about 20 to 30 per cent. of the weight of the seeds) melts off; the seeds are then comminuted in order to obtain the liquid fat. Or the seeds are comminuted at once, and the tallow and liquid fat boiled out together with water. The tallow obtained by the first method is white or greenish-white, inodorous, quite hard, and melts at  $104^{\circ}$  to  $105^{\circ}$  F.: it consists chiefly of palmitin. The fat obtained according to the other method forms a whitish or gray-white mass with a slight odor; it becomes yellow to brown by storing, shows a slightly acid reaction and congeals at about  $95^{\circ}$  F.

Chinese tallow, it is claimed, has been for some time used in England in the manufacture of candles and soap. About its

value for these purposes B. Lach\* says: "To obtain a serviceable product from this fat alone will very likely never succeed. Chinese tallow consists chiefly of tripalmitin, and, though it saponifies well under high pressure with lime, the resulting mass has an unsightly appearance and cannot be pressed. The press cloths become smeary and burst; besides, the fatty acids, even if successfully obtained, are and remain gray in spite of thorough washing. Subjecting the fatty acids thus obtained to distillation does not produce much better results. The distillate, though of a better appearance, is soft, cannot be pressed, and is serviceable only as an addition to other material.

"By combining Chinese tallow with other tallows quite different results are, however, obtained; it then acts almost like press-tallow. A mixture of an equal quantity of bone-fat and Chinese tallow yields a beautiful mass, which is readily pressed, and, when subjected to distillation, gives a distillate which also can be pressed. Worked by itself Chinese tallow yields ten per cent. of glycerin of  $28^{\circ}$  B., it being, however, advisable to repeatedly wash it with dilute sulphuric acid previous to the operation. The amount of dirt separated by this means is almost incredible.

"Commercial Chinese tallow varies very much in quality. The melting-point of the neutral fat, which is always much below that of the fatty acids, differs considerably, being sometimes as low as  $95^{\circ}$  F. The fatty acids generally drain off at  $131^{\circ}$  F.; with pale yellow tallow the temperature, however, rises to nearly  $140^{\circ}$  F., and with green tallow sinks to  $120^{\circ}$  F.

"Chinese tallow contains frequently considerable water, though in examining it, special attention should be directed towards dirt in the canisters, of which there is frequently found an astonishing quantity."

Under the name of "vegetable tallow" another vegetable fat is brought into market which is obtained, chiefly in Borneo, from various species of *Hopea*, especially *Hopea splendida* and *Hopea aspera*. The ripe fruits are piled up in a moist place and allowed to germinate a little. They are then dried in the sun until they

\* Chem. Zeitung, 1885. S. 941.



are brittle, when they are deprived of their shell and put into a rattan or bamboo basket suspended over boiling water. The arising steam renders the fruits soft and plastic. The fat is then expressed by squeezing the doughy mass in a cloth, and is poured into joints of bamboo, by which it receives the cylindrical form in which it is met with in commerce. By the natives the tallow is used for culinary and lighting purposes. It is largely exported to England, where it has been successfully employed as a lubricating agent, being very valuable for this purpose, far surpassing even olive oil. In Manilla it is employed in the manufacture of candles.

The tallow is white and hard and crumbling at an ordinary temperature, but softens at a moderate heat, and has a somewhat nutty taste and odor. According to Fielding, it remains hard up to a temperature of  $64.5^{\circ}$  F., softens between  $80.5^{\circ}$  and  $104^{\circ}$  F. to a pasty mass, and melts at  $112^{\circ}$  F. It dissolves in about half its weight of cold ether, is sparingly soluble in cold but freely in hot acetone; on cooling the greater portion is, however, re-separated. It dissolves in half its weight of chloroform, is readily soluble in oil of turpentine, and very freely in carbon bisulphide, and in hot benzene. It is also soluble in about 30 parts of cold and 20 parts of hot alcohol.

*Cocoa butter* is obtained as a by-product in chocolate factories by hot expression of the cocoa bean, the fruit of *Theobroma cacao*, L. In a fresh and pure state the fat is yellowish, but becomes nearly white with increasing age. It smells like roasted cocoa beans and keeps a long time without becoming rancid, and for this reason is much employed for cosmetic and pharmaceutical preparations. The specific gravity of fresh cocoa butter is 0.950 to 0.960, and that of old 0.947 to 0.950. The statements in regard to the melting-point vary between  $77^{\circ}$  and  $91.5^{\circ}$  F., the variations being, no doubt, chiefly due to the methods employed. Melted cocoa butter can, according to Rüdorff, be readily cooled to  $71.5^{\circ}$  F., and on congealing shows a permanent increase of temperature to  $82^{\circ}$  F. Cocoa butter gives a clear solution with ether, oil of turpentine, and chloroform. It consists, according to Traub, of the glycerides of oleic, palmitic, stearic, lauric, and arachidic acids, and is readily saponified. Its saponification

equivalent is 198 to 203, and its iodine degree 51.0. The separated fatty acids melt at  $125.5^{\circ}$  F., and congeal at  $123.5^{\circ}$  F.

Genuine cocoa butter is readily recognized by the taste, odor, and consistency. The principal adulterations are with wax, stearic acid, paraffin, and especially with beef kidney fat. All these substances are readily recognized by dissolving 1 part by weight of cocoa butter in 2 parts by weight of ether without the assistance of heat. Pure cocoa butter yields a clear solution, remaining so for at least one day; in the presence of more than 10 per cent. of tallow or wax the solution is more or less turbid, or forms a whitish sediment. The presence of tallow can also be detected by the odor evolved by an ignited wick soaked in the melted fat. An addition of stearin is recognized by boiling the suspected sample with solution of sodium carbonate and filtering after cooling; if stearin be present, stearic acid is separated by adding diluted sulphuric acid to the filtered fluid. Cocoa butter adulterated with paraffin feels soapy to the touch and shows a specific gravity of below 0.9. The paraffin can be quantitatively determined by the saponification test.

*Dika-oil, or wild mango-oil*, is obtained from the seeds of *Mangifera gabonensis*, a tree indigenous to the west coast of Africa. When fresh it is pure white and of a mild cocoa-like odor and taste, but when stored for some time it becomes yellow and rancid. It melts at from  $86^{\circ}$  to  $89.5^{\circ}$  F., contains the glycerides of lauric and myristic acids, and is readily saponified.

*Expressed oil of nutmeg, butter of nutmeg, or oil of mace* is extracted by heating and pressing from the kernel of the nutmeg, the fruit of *Myristica fragrans*. It is generally brought into commerce from the East Indies in oblong four-cornered pieces as thick as the arm and wrapped in plantain leaves. A large quantity of the fat is also prepared in Europe by pressure or extraction with absolute alcohol. The latter (*oleum nucistae germanicum*) is the cheaper and purer article. Expressed oil of nutmegs is reddish-yellow or yellowish brown-red (generally whitish and reddish-marbled), of the consistency of tallow, but more brittle and crumbling. It is unctuous to the touch, lighter than water, and enveloped in paper and ignited burns almost without smoke and with a very slightly sooting flame, and, when extinguished,

does not evolve the disagreeable odor of tallow. It has the strong aromatic odor of nutmeg, melts at from  $106^{\circ}$  to  $124^{\circ}$  F., has a specific gravity of 0.995, dissolves in 4 parts of boiling alcohol, with difficulty in cold alcohol, and more freely, but not completely in chloroform, ether, and benzene. According to Koller, 100 parts of it consist of 6 parts of volatile oil of nutmegs, 70 parts of myristin, 20 parts of olein, 1 part of butyrin, and 3 parts of an acid resin. Its iodine degree is, according to Hübl, 31.0.

Expressed oil of nutmeg is used in perfumery and in medicine. The principal adulterations are with lard, beef-marrow, tallow, and vaseline, besides coloring substances, such as turmeric, sanders-wood, etc. The first-named adulterations are, according to Hager, readily detected by triturating 1 part of the fat with about 50 parts of 96 per cent. alcohol, shaking, filtering, washing the myristin remaining upon the filter with alcohol, and drying with pressure between blotting-paper and exposure to the air. In the presence of the above-mentioned fats the filter and the press-paper show grease stains, and the myristin is not pulverulent to the touch, but fatty and smeary. An addition of turmeric is detected by the brown coloration with potash lye.

Besides from *Myristica fragrans*, the fat is also obtained from several other species of *Myristiceæ*. The fruit of *Myristica officinalis* yields the *becwiba tallow*, which resembles expressed oil of nutmegs, has a sharp, somewhat sour taste, melts at  $116.5^{\circ}$  F., and has a specific gravity of 0.956 at  $77^{\circ}$  F. It is saponifiable and yields a crumbling soap. The fruits of *Myristica Otoa* yield the *Otoa butter*. It is nearly colorless, of a butyraceous consistency, smells, when fresh, of nutmeg, melts at  $100.5^{\circ}$  F., and contains myristin, olein, and a non-saponifiable body, "otabite." *Pirola tallow* is obtained by boiling the peeled kernels of *Myristica sebifera* with water. It is yellowish, melts at from  $111^{\circ}$  to  $122^{\circ}$  F., is completely soluble in alcohol and ether, and only partially saponifiable. In America, England, and France it is used in the manufacture of candles.

*Olive-oil* is perhaps the oldest known and used for the purpose of making fine soaps, and possesses all the best characteristics for the purpose, yielding a firm white soap with an agreeable odor.



Olive-oil being consumed in large quantities for food, there is much care taken to produce a fine quality for this purpose, and the best of the first and second pressing is generally reserved for table use.

In the more southern regions the olives are comminuted together with the kernel, and the paste thus obtained is subjected to pressure; in more northern countries where the olive tree still thrives, but a regular yield cannot be calculated on, only the crushed fruit-flesh is pressed. The kernels remain whole, and after being freed from adhering fruit-flesh by boiling with water are comminuted and extracted. The oil thus obtained is generally brought into commerce under the name of "*olive-kernel oil*." It is dark green and thickly fluid, and readily deposits a considerable quantity of solid fat.

The oil chiefly used in the manufacture of soap is the so-called "*sulphur-oil*." It is prepared from rotten olives and residues from other pressings. It is thickly fluid, contains much stearin, and has a dark-green color. This oil being obtained by extraction with carbon bisulphide, benzene, etc., contains much vegetable mucus, albuminous substances, and a peculiar coloring substance.

The oil being very cheap but difficult to bleach, we recommend the following process: The oil is brought into an iron kettle or wooden vat and steam introduced through a perforated serpentine pipe placed upon the bottom. The green color of the oil gradually disappears, a slimy dark mass being deposited on the bottom of the vat. The oil is then allowed to rest a few hours, and after washing with two to three per cent. of caustic soda lye of 38° B. allowed to rest for a few days.

For manufacturers having no steam at their disposal the following process is recommended: Bring 300 pounds of salt water of 10° to 12° B. into a clean kettle and add 1000 pounds of sulphur olive-oil. Then start the fire under the kettle and allow the whole to boil slowly about three hours. A reddish-brown scum forms on the surface which has to be removed, and when this has entirely disappeared cover the kettle and allow the oil to stand in a warm place over night. The next morning bring the supernatant clear oil into a clean wooden vat. In the mean while

mix 20 pounds of peroxide of hydrogen with 3 pounds of sal ammoniac and heat the whole moderately. At the same time have in readiness 200 to 300 pounds of boiling salt water of  $5^{\circ}$  B.

One workman now crutches the oil uninterruptedly, while another pours the mixture of peroxide of hydrogen and sal-ammoniac in a fine stream over the oil. The crutching is continued until brownish streaks appear on the surface. The oil is then washed with the boiling salt water of  $5^{\circ}$  B. and allowed to rest over night.

The oil, though not entirely decolorized, has completely lost its green color and acquired a yellowish shade; it yields a fine, light soap.

Olive-oil is now seldom used alone, the soap becoming, when dry, too hard for general purposes. It is customary to add to it a certain quantity of hemp-seed, rape-seed, poppy-seed, or groundnut-oil; these oils, being slightly drying oils and producing a softer soap, qualify the olive-oil soap in its consistency.

Pure olive-oil is of a pale-yellow to greenish-yellow color and of a mild and agreeable taste. It dissolves sparingly in alcohol, but readily in  $1\frac{1}{2}$  to  $2\frac{1}{2}$  parts of ether and in 3 parts of acetic ether. The cold-pressed oils contain over 70 per cent. of olein; the remainder is palmitin, with some butine and stearin, and, according to Benecke, a very small quantity of cholesterin. The hot-pressed oils are richer in palmitin. The specific gravity of cold-pressed oils varies between 0.915 and 0.918 at  $59^{\circ}$  F., while that of the hot-pressed oils is frequently as much as 0.925. The latter deposit granular secretions at  $50^{\circ}$  F. and congeal at  $32^{\circ}$  F., while very fine cold-pressed oils become turbid only at  $35.5^{\circ}$  F., and separate palmitin and stearin at  $21^{\circ}$  F. The fatty acids separated from olive-oil melt at from  $71.5^{\circ}$  to  $79^{\circ}$  F. and congeal at from  $70^{\circ}$  to  $71.5^{\circ}$  F. The saponification equivalent of olive-oil is 191 to 192 and the iodine degree 81.6 to 84.6, and that of the separated fatty acids 86.1. The better qualities of olive-oil are frequently adulterated. An excellent means of recognizing pure olive-oil is the iodine degree, as nearly all oils used for adulteration show a higher degree.

Olive-kernel oil is readily soluble in alcohol and glacial acetic

acid, and has a specific gravity of 0.9202 at 59° F.; its saponification equivalent is 188.5 and its iodine degree 81.8.

*Sesame oil* is obtained from the seeds of two varieties of *Bigoniaceæ*, *Sesamum indicum*, L., and *Sesamum orientale*, L. The seeds are generally pressed three times, the first two pressures being cold and the third hot. The cold-pressed oils are used for table oils and the hot-pressed oils chiefly in the manufacture of soap. The taste of cold-drawn oil, while mild, is less agreeable than that of olive oil.

Sesame oil has a fine pale-yellow color and contains a small quantity of a resinous body, which can be extracted by frequent shaking with glacial acetic acid; it has a specific gravity of 0.922 to 0.924 at 59° F., and congeals at 23° F. to a yellowish-white, transparent, somewhat unctuous mass of the consistency of palm-oil, which, in this state, it very much resembles. The oil consists chiefly of olein and very little palmitin, stearin, and myristicin. Its saponification equivalent is 190 and that of the separated fatty acids 199.3, the melting-point of the latter being at from 77° to 79° F. and their congealing-point at 72° F. The iodine degree of the oil is from 102.7 to 106, and that of the separated fatty acids from 108.9 to 111.4.

In the manufacture of soap only the hot-pressed oil or that obtained from seed of poor quality is used, and also the oil extracted by means of carbon bisulphide or benzene. For bar-soap only the nearly white, thick sedimentary portion deposited in storing sesame oil for some time, and containing much stearin or palmitin, can be used. The liquid portion (as much as 30 per cent.) is employed as an addition to palm-kernel oil, cocoanut-oil, tallow, and palm-oil, with which it yields good soaps.

Sesame-oil prepared from mouldy seed has generally a very disagreeable odor, which can, however, be removed to a great extent by boiling the oil upon water with steam, then bringing it into a large reservoir and allowing it to stand. In cooling, much solid fat suitable for bar-soap is separated.

The sedimentary portion of sesame-oil is readily saponified, behaving thereby in a manner similar to lard. In boiling with caustic soda lye, the oil is gradually added to the boiling lye, 200



parts by weight of lye of  $15^{\circ}$  B. being generally allowed for 100 parts by weight of the oil.

*Peanut or groundnut-oil.*—This oil is obtained from the fruit of *Arachis hypogæa*, or the peanut, a legumine plant. This small creeping plant is indigenous to South America and the coasts of southern Africa and Asia. Since the latter part of the last century it has been cultivated in our Southern States, and in Italy, Spain, and the southern parts of France.

The nuts are generally pressed three times, the first cold pressure yielding a nearly colorless oil of an agreeable taste and odor, which is used as fine table oil. The comminuted nuts are then sprinkled with water and subjected to a second cold pressure, which yields an oil also fit for table use, but chiefly employed as an illuminant. The residue is subjected to a third warm pressure, and yields a yellowish-brown oil of a less agreeable taste and odor. It is chiefly used in the manufacture of soap. One hundred pounds of peanuts generally yield 30 to 34 pounds of oil.

Peanut-oil is somewhat more thinly fluid than olive-oil and contains the glycerides of oleic, palmitic, hypogæic, and arachidic acids. The specific gravity of the finest fresh oil is 0.916 at  $59^{\circ}$  F., while that of the second and third pressures is as much as 0.920. Peanut-oil belongs to the non-drying oils; it is quite durable and does not very soon become rancid. It becomes turbid at  $37.4^{\circ}$  F., congeals at  $26.5^{\circ}$  F., and solidifies entirely at  $19^{\circ}$  F. Its saponification equivalent is from 191.3 to 191.6; its iodine degree, according to Hübl, 103, and, according to Moore, 87.3. The separated fatty acids melt at  $82^{\circ}$  F. and congeal at  $75^{\circ}$  F.; their iodine degree is from 95.5 to 96.9.

Peanut-oil is used in the manufacture of grain-soap, half-boiled soap, and soft soap. Lyes of not less than  $18^{\circ}$  B. are used for saponification. The oil in many respects resembles cotton-seed oil, but has the advantage that the soaps do not show yellow stains. Two parts of peanut-oil and 3 parts of palm-kernel oil, boiled directly with caustic soda lyes, yield faultless wax grain-soap. Beautiful half-boiled soaps are obtained from 70 per cent. of palm-kernel oil and 30 per cent. of peanut-oil, with caustic soda lye. It is best to boil in a direct way, using

pure caustic lye of about  $24^{\circ}$  B., and afterwards reducing causticity by solution of soda or common salt.

As regards soft soaps, peanut-oil is especially adapted for the manufacture of so-called silver soap, or elaïdin soap, being preferable for this purpose to cotton-seed oil. For cold-stirred soaps the use of peanut-oil is of great advantage. A soap, for instance, of 70 pounds of cocoanut-oil, 30 pounds of peanut-oil, and 60 pounds of caustic soda lye of  $36^{\circ}$  B. is superior in appearance to a soap in which tallow is used instead of peanut-oil. Peanut-oil is also well adapted for the preparation of soaps for a basis of fine toilet-soaps. For transparent glycerin soaps peanut-oil cannot, however, be substituted for tallow, as the soaps would become too soft.

*Castor-oil* is obtained from the seed of *Ricinus communis*, L., by pressing in the cold way, or, as it is frequently done in this country, by slightly roasting the seeds before pressing. The oil for medicinal purposes is obtained from the heavy, sound seeds and filtered. A second quality of oil is made from imperfect seed and the residue from the filtration of the first quality. This oil is largely used in the manufacture of toilet-soaps. A third quality of oil, prepared from the sediment, residues, etc., is dark and frequently of a strong and disagreeable odor. It is used as a lubricant and in Turkey red dyeing.

Castor-oil is colorless or slightly yellowish, of a mild, with an acrid after-taste and a slight though not disagreeable odor. It is chiefly composed of the glyceride of ricinoleic acid and a small quantity of stearin and palmitin. Its specific gravity varies between 0.95 and 0.97 at  $59^{\circ}$  F. At  $32^{\circ}$  F. it deposits a white stearin-like fat, and at  $1.4^{\circ}$  to  $-0.4^{\circ}$  F. congeals to a yellowish, transparent mass. American castor-oil frequently separates stiff fat at  $42.5^{\circ}$  F., and congeals at from  $14^{\circ}$  to  $10.5^{\circ}$  F.

The saponification equivalent of castor-oil is, according to Valenta, from 181.0 to 181.5; its iodine degree is 84.4, and that of the separated fatty acids 86.6 to 83.3. The fatty acids melt at  $55.5^{\circ}$  F., and congeal at  $37.5^{\circ}$  F.

The behavior of castor-oil during saponification is similar to that of cocoanut-oil, it being readily saponified by stirring together with strong soda lye. The resulting soap is very white, amor-

phous and transparent, and quite hard, even if it contains as much as 70 per cent. of water, but gives scarcely any lather. It has also the property of dissolving in pure, cold water without rendering it turbid or opalescent. It, therefore, seems that sodium ricinoleate is not decomposed by water into an acid salt and basic sebate.

Castor-oil is chiefly used in the manufacture of transparent soaps.

*Cotton-seed oil* is obtained from the seeds of the cotton plants, *Gossypium herbaceum*, *G. arboreum*, *G. barbadense*, and other varieties. The seeds having been screened from all dust and foreign substances are freed from adhering cotton by passing them through a machine similar to a gin, only with teeth placed more closely together. They are then brought into the huller, which consists of a cylinder armed with steel blades, and surrounded about two-thirds way by a concave box also armed with corresponding knives. The cylinder revolves at great speed, and as the seeds are forced between the knives the pericarps or hulls are broken and forced from the kernels. The mass then falls into a large revolving sieve. The kernels, many of which are broken into fine pieces, pass through the meshes of the sieve and the hulls are carried away. The clean seeds are now crushed between rolls and the resulting meal heated in a pan until the water it contains is evaporated. The hot meal is then placed in wedge-shaped bags of woolen duck, each holding sufficient for a cake. The bags are placed between the sides of wrappers formed of woven horse-hair backed with corrugated leather to facilitate the escape of the oil, and subjected to pressure in a hydraulic press. Some modern presses are so arranged as to do away with the expensive bags and horse-hair mats.

The crude oil, as it comes from the press, is reddish or dirty yellowish, thickly fluid, and has a specific gravity of 0.922 to 0.930 at 59° F. It commences to separate palmitin at below 50° F., and congeals between 28.5° and 26.5° F.

The process of refining the crude oil, which was kept secret for a long time, consists essentially in a treatment with alkaline carbonates and caustic alkalies.

The tank used is of iron; it is provided with a mechanical



agitator, and its capacity sufficiently exceeds that of the charge of ten tons' weight of crude oil and thirty hundred weight of caustic soda lye of  $10^{\circ}$  to  $12^{\circ}$  Twadell. The lye, at the temperature of  $60^{\circ}$  F., is fed slowly by perforated pipes extending over the surface of the oil and distributing uniformly. As the agitation proceeds, the lye and oil, which are both cool, mix, and the latter gradually becomes full of black, flocculent particles of soap, caused by the partial saponification of a portion of the oil by the caustic soda lye. The agitation is continued for about half an hour; and at the end of that time a portion is taken out and allowed to stand. If the soapy particles precipitate and the oil is found nearly deprived of color, the operation is then terminated. If not, the agitation is continued, more lye being added until the desired discoloration is obtained. The charge of oil is then allowed to stand for twelve or fifteen hours until the "mucilage," or partially saponified portion of the oil, with the liquid excess of lye used, has settled away. The clear oil is then run off from the brown sediment called "soap-stock," and the refining completed by washing and bleaching.

The soap-stock may be either converted into hard soap, or, by addition of milk of lime, be deprived of any albuminous matter, bleached with chloride of lime, and, by treatment with mineral acid, the refined oil mingled with fatty acids obtained.

Refined cotton-seed oil is of a pale straw color, has a pure nutty taste, and a specific gravity of from 0.923 to 0.928 at  $59^{\circ}$  F. Its saponification equivalent ranges from 191 to 196.5, and that of the separated fatty acids from 110.9 to 111.4. The iodine degree of the oil is from 106 to 108.7, and that of the fatty acids from 110.9 to 111.4. The separated fatty acids melt at from  $95.5^{\circ}$  to  $101^{\circ}$  F., and congeal at from  $95^{\circ}$  to  $100.5^{\circ}$  F.

In the United States the crude cotton-seed oil is used for lubricating purposes, as a substitute for linseed-oil in the manufacture of varnish and in the fabrication of soap; the refined oil is employed as a table oil, in the manufacture of soap, and largely for the adulterations of other oils.

The crude oil saponifies with greater ease than the refined oil; this is very likely due to the former containing free fatty acids, while the latter is neutral in consequence of the treatment with

lyes in refining. The refined oil saponifies with difficulty even with strong lyes, but readily in combination with easily saponifiable fats, such as palm-kernel oil, cocoanut-oil, etc. By itself it can only be gradually brought to complete saponification by weak lyes; the resulting soap has, however, the disagreeable property of being difficult to salt out, and of not completely yielding the excess of water, even with a large addition of common salt. Such soap is so soft and smeary that it can be worked with a shovel, and, on pressing in the hand, yields water. The fresh soap is white, but becomes yellow after drying, and has a peculiar bad odor.

In Europe refined oil only is used in the manufacture of soap. It is employed for hard and soft soaps; however, never by itself, but always in combination with other fats on account of its difficult saponification and the disagreeable odor acquired by pure cotton-seed oil soaps on storing. Soaps manufactured from a large percentage of cotton-seed oil in combination with other fats also show this disagreeable odor; it can, however, be removed to some extent by boiling the oil with soda lye of 25° B. before use.

Commercial refined oil is of a sufficiently pale color for most soaps for which it is used. Where a lighter color is desired the object can be attained by treating the oil with caustic soda lye or with potassium chromate and hydrochloric acid.

For hard soaps cotton-seed oil is generally used in combination with palm-kernel oil and cocoanut-oil, it possessing the property of making soaps from these oils soft and delicate. At one time cotton-seed oil was very much liked for smooth white-grain soaps, but the soap-boilers who were first in raptures over the fine results attained, had soon cause for regret when yellow stains made their appearance. These yellow stains are due to a yellow, non-saponifiable, oleaginous body in the oil, which is not destroyed by refining with lye and not completely removed by repeated refining. In France and Italy considerable quantities of cotton-seed oil are worked in combination with peanut-oil into Marseilles soap, though not to the advantage of the product.

Cotton-seed oil is also much used for soft soaps. On account of its comparatively large percentage of palmitin it cannot be

used in winter for smooth, transparent soft soaps, as they readily become turbid; in summer it is, however, very suitable for such soaps, they being more solid than with the use of pure linseed-oil.

For smooth yellow and smooth white soft soaps cotton-seed oil is well adapted and can be used by itself. For soap with a silver lustre, so-called silver or elaïdin soap, it must be previously bleached.

It is also used for grained soft soaps, though great care is required in not allowing any soda to reach the soap and using only the best high-graded potash.

The property of cotton-seed oil separating palmitin at a few degrees above  $32^{\circ}$  F., has been utilized in the United States to obtain an oil containing little palmitin, and, therefore, better adapted for the adulteration of olive-oil than the natural refined oil. The separated solid fat is of a lardaceous consistency and is brought into commerce under the name of "cotton-stearin" or "vegetable stearin." According to Muter,\* it has a specific gravity of 0.9115 to 0.912 at  $100.5^{\circ}$  F., is completely soluble in ether and hot absolute alcohol, and by saponification yields 95.5 per cent. of fatty acids belonging to those insoluble in water. Though the fat becomes completely liquid only at  $89.5^{\circ}$  F., the melted fat does not recondense after cooling, but forms a yellow oil, which only acquires its original consistency by long cooling at about  $40^{\circ}$  F.

Cotton stearin shows the same disagreeable properties in saponifying as cotton-seed oil, while soaps prepared from it show yellow stains and acquire a disagreeable odor on storing.

*Almond-oil.*—The fat almond-oil found in commerce is generally obtained from small bitter almonds, peach and apricot kernels, and fragments of sweet almonds. It is limpid, thinly fluid, slightly yellowish, nearly inodorous, of a pleasant, mild taste, and belongs to the non-drying oils. Its specific gravity is 0.915 to 0.920 at  $59^{\circ}$  F. The actual almond-oil congeals at  $4^{\circ}$  F., peach-kernel oil at  $0.4^{\circ}$  F., and apricot-kernel oil at about  $7^{\circ}$  F.

\* Seifenfabrikant, 1882. S. 411.



The best qualities of almond-oil are used for medicinal purposes and for the adulteration of table oil. The inferior qualities are employed for technical purposes, and are especially in much demand for the manufacture of toilet soaps. A cocoa-soap of three-quarters cocoanut-oil and one-quarter almond-oil is very hard and firm and an excellent toilet soap.

Besides with peach-kernel and apricot-kernel oils, almond-oil is chiefly adulterated with poppy-oil, sesame-oil, peanut-oil, beech-nut oil, and walnut-oil.

*Linseed-oil* is obtained from the seeds of the flax-plant, *Linum usitatissimum* L., by pressure and the aid of heat. Ripe seeds contain 30 to 35 per cent. of oil and those not thoroughly matured less. Commercial seed yields on an average 22 per cent. of oil. Cold-drawn linseed-oil is nearly colorless; hot-pressed oil has a golden-yellow color changing to brown with age.

Linseed-oil has a peculiar odor and possesses the most drying properties of all known oils. On exposure to the air it eagerly absorbs oxygen, soon becoming rancid and thickly fluid; in a thin layer it dries to a neutral body insoluble in ether. It has a specific gravity of 0.930 to 0.935 at 59° F., and congeals to a solid yellow mass at -16.5° F. The fatty acids separated from linseed-oil melt at from 52° to 62.5° F., and congeal at about 55.5° F. The saponification equivalent is 189 to 195, the iodine degree 155 to 158, and the iodine degree of the separated fatty acids 155.2 to 155.9. Linseed-oil is soluble in 5 parts of boiling and in 40 parts of cold alcohol and in 1.6 parts of ether. It consists of 10 per cent. of palmitin and myristin, 10 per cent. of olein, and 80 per cent. of linolein, the glyceride of linoleic acid.

Many complaints have been lately heard about the adulteration of linseed-oil. The surest way of testing the oil as to its purity is the determination of the iodine degree, that of linseed-oil being the highest of all known oils. A very simple test is to expose the oil to a low temperature; oil which separates solid fat at a few degrees below 32° F., or congeals, is not pure linseed-oil.

Linseed-oil is largely employed in the manufacture of soap in Europe, particularly in Germany, where at times it is so low in price as to be the cheapest fat to use. In combination with other

fats it is well adapted for various kinds of soap, and if properly used gives very satisfactory results. Potash soaps from pure linseed-oil do not freeze even on exposure to the coldest weather occurring in this country. For summer soft soap it is recommended not to use linseed-oil by itself, but to add some oleic acid or cotton-seed oil. For the transparent, pale soft soaps so much in demand at the present time the linseed-oil has to be bleached. A pale oil may be used without bleaching by thoroughly boiling in a lye of  $23.5^{\circ}$  to  $26.5^{\circ}$  B. This must, however, be determined by experiment, because a pale linseed-oil cannot be well defined. An oil may appear pale and clear, but when it is boiled the soap rapidly becomes darker in color, while another oil of the same appearance will give a soap that will remain light.

A dark brownish linseed-oil, which is not decolorized by lye, cannot be improved by the use of bichromate and acid. Oil of a greenish tinge generally bleaches very light with bichromate and acid. Generally speaking, linseed-oil saponifies with ease. Oil bleached with lye, being entirely neutral, is somewhat more difficult to saponify than crude oil or that bleached with bichromate and acid. It further deserves attention that a thinly-fluid oil like linseed-oil requires lyes of greater causticity than an oil containing more solid constituents, as otherwise the soap turns out too soft. An advantage of linseed-oil is that many distilled oleins, which by themselves do not yield a serviceable soap, can be worked in combination with it.

When, a few years ago, there was an extraordinary advance in the price of all fats, while that of linseed-oil remained at the old figure, the latter was also used for the fabrication of bar-soaps, half-boiled soaps, and resin soaps. The soda soaps from linseed-oil have the property that, like tallow and olive-oil soaps, they can stand but little common salt. Soaps of about 20 per cent. linseed-oil and 80 per cent. palm-kernel oil, when fresh, were beautiful, but complaint was made that on storing for some time they showed dark stains and acquired a bad odor even in a higher degree than soaps from cotton-seed oil. As linseed-oil does not contain non-saponifiable constituents like cotton-seed oil, it would seem that these defects must be due to incomplete saponification. Soap-boilers very frequently do not take into consideration the

fact that the last portions of most fats and oils saponify with extraordinarily great difficulty, and that a soap containing non-saponified fat readily turns rancid.

A mixture of 20 parts linseed-oil, 20 parts bone-fat, and 60 parts palm-kernel oil will give a very good winter soap, which will form a larger grain with more certainty than a pure linseed-oil soap. In this case the linseed-oil and bone-fat are first boiled to a curd with a lye of  $12^{\circ}$  B. ; in this way the two fats are simultaneously bleached. If water-glass is also to be employed, 100 pounds of pure soda lye of  $25.5^{\circ}$  B. and 15 pounds of water-glass are put in the kettle for each 100 pounds of palm-kernel oil. The curd is added to this and the whole well boiled, after which the palm-kernel oil is added, the soap being from time to time crutched until it boils well in the kettle.

The nature of linseed-oil permits the preparation of exceedingly pure soaps. The linseed-oil soaps do not resist the action of the air very well, and should therefore be consumed shortly after being made.

Resin soaps manufactured with the assistance of linseed-oil readily become too soft.

*Cameline-oil* or *German sesame-oil* is obtained by expression from the seeds of the gold-pleasure, *Camelina sativa*, Cez., or *Myagrum sativum*, L., natural order *Cruciferae*. It is golden-yellow, has slight drying properties, and a peculiar taste and odor. Its specific gravity is 0.925 to 0.930 at  $59^{\circ}$  F. ; it congeals at  $-0.4^{\circ}$  F. Cameline-oil, being produced in comparatively small quantities, is of but little importance, though it is well adapted for the fabrication of soft soaps which do not freeze even on exposure to great cold. Barrel soaps from cameline-oil can scarcely be kept in summer, as they melt below  $68^{\circ}$  F.

*Niger-oil* is obtained from the seeds of *Guizotia oleifera*, D. C., cultivated in India. Large quantities of seed are shipped from Bombay to England, where they are expressed. The oil is of a yellowish color and a mild and nutty taste and odor. It has a specific gravity of 0.924 at  $59^{\circ}$  F., thickens at  $17.5^{\circ}$  F., forms a transparent yellowish mass at  $14^{\circ}$  F., and a solid whitish mass at  $5^{\circ}$  F. In England it is much used in the manufacture of soap.



*Madi-oil* is obtained from the seeds of *Madia sativa*, Mdt., natural order *Compositæ*, indigenous to Chili. Experiments in cultivating the plant in France and Southern Germany proved a failure. The oil is dark yellow and has a peculiar, though not disagreeable, odor and taste. Its specific gravity is 0.928 to 0.935; it congeals, according to Winckler, at between 14° and 12° F., and, according to Riegel, at —13° F. With soda lye it yields a firm, inodorous soap.

*Hemp-seed oil* is expressed from the seeds of *Cannabis sativa*, L. Large quantities of the oil are brought into commerce from the Russian Baltic provinces. It has a strong odor and a sickly taste. When fresh it is of a greenish-yellow color, but becomes brown-yellow with time. Its specific gravity is 0.925 to 0.931 at 59° F.; it thickens at 5° F. and concretes at —16.5° F. The melting-point of the separated fatty acids is at 66° F. and their congealing-point at 59° F. The saponification equivalent of the oil is, according to Valenta, 193.1; the iodine degree, according to Hübl, 143; and that of the separated fatty acids, according to Morawski and Demski, 122.2 to 125.2. In boiling alcohol hemp-seed oil dissolves in all proportions; of cold alcohol it requires 30 parts. A solution of 12 parts in boiling alcohol, on cooling, separates stearin. Hemp-seed oil has strong drying properties. It was formerly much used in the fabrication of soft soaps which have a dark green color and can be submitted to intense cold without solidifying. The green soft soaps found at present in commerce are mostly artificially colored linseed-oil soaps.

*Sunflower-oil*.—This oil, obtained by expression from the seeds of several species of *Helianthus*, is chiefly brought into commerce from Russia. It is limpid, pale yellow, dries slowly, and, when cold-drawn, has an agreeable odor and mild taste. Its specific gravity is 0.924 to 0.926; it congeals at about 3° F. The separated fatty acids melt at 73.5° F. and congeal at 62.5° F.

At the present time the larger part of the oil is consumed in Russia itself, where the cold-drawn oil is used as a table-oil and the hot-pressed in the manufacture of varnish and soap. Considerable quantities were formerly exported and were readily

taken by soap-boilers, the oil yielding good bar soap and soft soap.

*Poppy-seed oil* is extracted by expression from the seeds of *Papaver somniferum*. It has a slight odor and mild taste; is nearly colorless or pale golden-yellow and limpid. The oil of the second pressure is darker. Poppy-seed oil has a specific gravity of 0.924 to 0.937 at 59° F. and congeals at  $-0.4^{\circ}$  F. The separated fatty acids melt at 69° F. and congeal at 62° F. The saponification equivalent is 192.8 to 194.6; the iodine degree 134 to 136.

Poppy-seed oil is chiefly used as a table-oil and in oil painting. On account of its high price it is not much used in the fabrication of soap; the thick sedimentary oil serves for the fabrication of soft soaps.

*Colza-oil*.—Under this name are known the oils obtained from different varieties of *Brassica*. Their properties agree in all principal points, the specific gravity varying between 0.9128 and 0.9175 at 59° F. and the congealing-point between 28.5° and 14° F. The saponification equivalent is 177 to 179, the iodine degree 100 to 103.6, and that of the separated fatty acids 96.3 to 99.02. The colza-oils consist chiefly of the glycerides of oleic, stearic, and brassic acids, and contain, according to Allen and Thompson, one per cent. of non-saponifiable substance. Their very low saponification equivalent is due to the percentage of brassic acid. The color of colza-oils is pale or dark brown-yellow, the product of the first pressure being somewhat paler than that of the second pressure. When fresh the oils are almost inodorous, but acquire a peculiar odor by age. They have an acrid taste, especially those of the second pressure.

Before the introduction of petroleum colza-oils were largely used for illuminating purposes, but at present they are chiefly employed as lubricants. They are seldom used in the manufacture of soaps, with the exception of the thick sedimentary oils which are frequently utilized for soft soaps. They saponify with difficulty, and the soft soaps prepared from them break up at a moderate cold. With soda lye colza-oils yield a poor, crumbling soap.

*Utilization of sedimentary oils.*—Large lots of sediment from oil reservoirs are frequently offered to soap-boilers. These sedimentary oils generally constitute a dark and smeary mass, and are suitable for the fabrication of soft soaps and bar soaps which are very valuable for fulling. The oils can also be used for resin soaps, the strong odor adhering to them covering to some extent that of the resin. The oils are worked in various methods; some boil them to grain and add a small quantity of the resulting soap to each boiling of soap, while others add a small quantity of the sedimentary oil directly to the soap-stock, which can be very well done, for instance, with half-boiled soap. It depends, however, more or less on whether the sedimentary oil is comparatively pure and not too old. Another method yielding an excellent product consists in boiling the sedimentary oil upon strong salt water until the clear oil free from scum floats on top. One thousand pounds of the oil are brought into an open kettle, together with 800 pounds of water and 60 pounds of salt, and boiled 10 to 12 hours. The kettle is then allowed to stand uncovered for 24 hours, when the clear oil on the top is drawn off. Beneath the clear oil is a layer of slime mixed with hulls of seed, etc. The oil retained by this is gradually separated by filling petroleum barrels half full with this slime, adding salt water, and after covering the barrels exposing them to the sun. This process is still better if executed with steam.

### *Fatty Acids and Resin.*

The use of fatty acids in the fabrication of soaps is based upon a much more simple chemical process than that which takes place in the saponification of neutral fats, as no previous splitting into glycerin and fatty acids is required, and the fatty acids possess the property of expelling the carbonic acid from alkaline carbonates and combining with them to soap. With the use of fatty acids the preparation of lyes might, therefore, be saved and saponification effected with solutions of alkaline carbonates, if it were not for the strong effervescence produced by the escape of the liberated carbonic acid; hence the lye can only be very gradually added to the fatty acid in the kettle, as otherwise a boil-



ing over might easily take place. For this reason lyes partly caustic, though less so than those employed for neutral fats, are always used without fear of suffering a loss of alkaline carbonates. While in working neutral fats they are first brought into the kettle and the lye is gradually added, the process is the reverse with fatty acids, *i. e.*, the fat is added to the lye. If saponification were effected as with neutral fats, by the gradual addition of lye, the latter would be at once fixed by the fatty acid, and solid lumps would be formed which could only later on be dissolved with difficulty by boiling with an excess of lye.

The use of fatty acids in the fabrication of soap is perhaps as old as the manufacture of stearin. In the patent granted in 1825 to Gay-Lussac and Chevreul, for the separation of fatty acids and their application to the manufacture of candles, it is stated: "The liquid bodies separated are to be converted into soap."\*

The crude oleic acid of stearin manufactories was the only fatty acid used in the fabrication of soap until the fatty acids regained from the wash-waters of cloth mills and other textile industries were brought into commerce under the name of "fuller's fat." The high price of glycerin a few years ago induced several stearin manufacturers to saponify neutral fats in order to obtain the glycerin and to sell the fatty acids to the soap-boiler. In this manner fatty acids of palm-oil, palm-kernel oil, olive-oil, and bone-fat came into commerce. They were no doubt prepared by saponifying the fats by De Milly's autoclave apparatus (see page 48) and distilling the separated fatty acids with superheated steam to give them a beautiful white appearance. Since the neutral fats contain at the utmost 95 per cent. of fatty acids, the latter, when pure, must give a correspondingly higher yield than the neutral fats from which they are separated. In regard to their ability in absorbing water, salts, etc., soaps from fatty acids show the same behavior as the soaps from the corresponding neutral fats. Another fatty acid—the so-called "*rapoleïn*"—has been recently offered to soap-boilers. It is a clear, dark red, nearly inodorous oil, and is very likely prepared from residues

\* Brevets d'invention 41, p. 392.

obtained in refining colza-oil. It is well adapted, either by itself or in combination with linseed-oil, for the fabrication of summer soft soaps, though only where no objection is made to their brown color.

Of the above-mentioned fatty acids it will be only necessary to give a full description of oleic acid, also called olein or clain, and of fuller's fat.

*Oleic acid.*—In the manufacture of stearin the fat, as previously mentioned, is saponified for the separation of the glycerin, and the resulting mixture of fatty acid is separated by pressure into a solid and liquid portion. The cakes obtained by the first cold pressure are again subjected to a second warm pressure. The resulting solid white mass is known as "stearin," though scientifically the term is incorrect, since the product is not the glyceride of stearic acid, but essentially a mixture of stearic and palmitic acids.

The liquid portion running off by the first cold pressure is "oleic acid," which, however, contains a considerable quantity of stearic and palmitic acids, and, with previous autoclave saponification, some dissolved neutral fat. By long storing in a cool cellar, or better, by cooling with a cold-air machine, the greater portion of the solid fatty acids separates out and is extracted by a filter-press or other filtering arrangement. The finally resulting clear, oleaginous fluid forms the "oleic acid." In commerce a distinction is made between "saponified" and "distilled" oleic acid, the latter being less suitable for soap-making purposes than the former. To fully understand this distinction we must bear in mind that in the fabrication of stearin (fully described in Chapter II.) several methods are employed for the saponification of the fats, chiefly the so-called autoclave process and acid saponification and subsequent distillation. Saponification by means of lime, which is the oldest method, has been entirely abandoned, chiefly on account of the large consumption of sulphuric acid required for the decomposition of the lime-soap formed. The principal source of saponified oleic acid at the present time is the autoclave process, *i. e.*, saponification under pressure in a closed vessel with 2 to 4 per cent. of lime. For the separation of the latter sulphuric acid is also used, but less lime being employed

than in ordinary saponification by means of lime, correspondingly less sulphuric acid is required for the decomposition of the lime-soap.

Acidy saponification is effected by bringing the heated fat for a shorter or longer period in contact with more or less concentrated sulphuric acid and then boiling for some time with water and steam. After allowing the whole to rest for a while, the mixture of fatty acid separates on the top. The aqueous layer beneath, which contains the glycerin and sulphuric acid in solution, is then removed and the fatty acid mixture again boiled with water and steam. The whole is then allowed to rest for some time, after which the fatty acid collected on top is lifted off and submitted to distillation by means of superheated steam. By the repeated boiling with water and steam the sulphuric acid is generally completely removed, but any trace of it remaining is eventually expelled by the subsequent distillation.

If, therefore, the opinion held by many soap-boilers, that the poor quality of distilled oleic acid is due to a content of sulphuric acid, may be regarded as erroneous, the question remains, what has brought distilled oleic acid into disrepute? It was formerly generally held that the defects were due to distillation, as oleic acid could not be distilled without suffering decomposition. This opinion has, however, been refuted by Bolley and Bergmann's investigations. They found that oleic acid distils over unaltered in a current of steam of  $482^{\circ}$  F., but that products of decomposition make their appearance at a higher temperature. In many stearin manufactories distillation is carried on at too high a temperature, they being forced to do this on account of the fat not being completely saponified and containing too much neutral fat. The poor quality of distilled oleic acid may, therefore, be due to products of decomposition formed under the influence of too high a temperature, and also to a content of non-saponifiable products originating from the treatment of the fat with sulphuric acid.

Formerly, when acidy saponification was generally carried on by allowing large quantities of concentrated sulphuric acid to act a long time upon the fats, a portion of the oleic acid was always destroyed, while the remainder was so changed as to render it almost unfit for the manufacture of soap. Besides, the oleic acid



always contained a considerable quantity of non-saponifiable, paraffin-like combinations (hydrocarbons). But this has been changed since experience has taught that fats can be saponified by leaving them a very short time in contact with concentrated sulphuric acid and completing saponification by boiling with water acidulated with sulphuric acid. Stearin manufacturers using this process of acid saponification now furnish oleic acid suitable for soap-making purposes. It may be even said that the greater part of the so-called saponified oleic acid at present found in commerce is actually distilled, as many factories saponifying in autoclaves subsequently slightly acidulate the fatty acids obtained and submit them to distillation. With many samples of oleic acid it is, therefore, almost impossible to decide by which process of saponification they originated, while others by their acrid odor at once indicate their production by distillation.

The best means of examining oleic acid is to test it in regard to its saponifying ability, *i. e.*, to determine how much non-saponifiable substance it contains. This is done in exactly the same manner as examining for mineral-oils or resin-oils (see page 102). Saponify the oleic acid with alcoholic soda lye, mix the resulting soap with sand, evaporate the alcohol in a water-bath, wash the residue with petroleum spirit, and remove the latter from the extract by evaporating at 122° F. The residue gives the non-saponifiable, paraffin-like combinations.

A method somewhat less exact, but sufficiently accurate for technical purposes, is by directly determining by titration the quantity of saponifiable substance. Oleic acid requires 19.87 per cent. of potassium hydroxide, and as palmitic acid requires 21.89 per cent. and stearic acid 19.73 per cent., we will not be far wrong in assuming 20 per cent. for oleic acid. Mix 1 gramme of oleic acid with 20 to 25 cubic centimetres of alcohol, color the mixture yellow with a few drops of phenol-phthalein, and titrate with potash lye containing 20 grammes of potassium hydroxide to the litre, until the appearance of a red coloration. With absolutely pure oleic acid, 1 gramme would almost exactly require 10 cubic centimetres of this lye for saturation. Such oleic acid is, however, not found in commerce, it generally containing at the utmost 97 or 98 per cent. of saponifiable substance.

It would therefore require 9.7 or 9.8 cubic centimetres of the lye, so that 0.1 cubic centimetre of the latter always corresponds to 1 per cent. of oleic acid. Oleic acid containing less than 95 per cent. of saponifiable substance cannot be designated as good, though it is found in commerce with more than 20 per cent. of non-saponifiable substance.

Oleic acid is used for grain-soap, smooth elain soap, and, in connection with linseed-oil, for transparent soft soap. The latter being brown, the demand for it has, however, much abated since the introduction of light-colored soft soaps. Oleic acid is much liked for soaps to be used in the textile industries. Hard oleic acid soaps are frequently used in place of olive-oil soaps.

The fatty acid mixture separated by hot pressure in stearin factories is of a lardaceous consistency and is generally known as "soft fat" or "margarin." It is generally added to crude fatty acids before subjecting them to pressure, though it is sometimes worked into soap. It yields excellent grain-soaps, but is especially adapted for smooth elain soap, so-called silver soap.

Under the name "white elain," a white fatty acid of lardaceous consistency is brought into commerce from Holland. A few years ago, when the price of tallow ruled very high, it was frequently employed, in connection with cocoanut-oil and palm-kernel oil, in the manufacture of white grain-soap. This white elain must not be confounded with the "solid white olein" which will be referred to under fuller's fat.

*Fuller's fat.*—The oldest method of recovering the fatty substances from the soap-waters of cloth manufactories, spinning establishments, dye-houses, etc., consisted in leading the water running from the wash-tubs into special cisterns, mixing it there with milk of lime, then letting it settle until it had cleared off. After removing the supernatant liquid, the slimy sediment was taken out, strained through coarse canvas for removing sand, hair, etc., and well dried. The slime having assumed a doughy consistency, it was moulded into pieces of the size of half a brick and dried in the air. The dried pieces, called "suinter," from the French word "suint" (wool sweat), were then distilled in retorts for the fabrication of illuminating gas. This method of

recovering the fat has, however, been abandoned, as it had many defects, and the process with sulphuric acid substituted for it.

The soap-water is conducted into reservoirs of pine wood. Such a reservoir is about 9 feet long,  $5\frac{1}{2}$  feet wide, and 5 feet deep, and holds about 1850 gallons. Sulphuric acid is added, and, in order to hasten separation, steam is introduced for from 1 to 2 hours. The quantity of sulphuric acid to be used depends chiefly on the amount of alkali in the soap-water; a slight excess is, however, always allowed, since by it a quicker and more complete separation, and, in consequence thereof, a more compact mass is obtained. On an average, 55 pounds of sulphuric acid of  $66^{\circ}$  Baumé suffice for a complete decomposition of 1850 gallons of soap-water, and will yield, according to the time allowed for draining off in the filtering basins, from 430 to 450 pounds of fatty matter. The filtering vessels consist of baskets lined with coarse hemp cloth. When the caseous, doughy mass is sufficiently drained off and has acquired the plastic consistency required for forming it into press-cakes, it is wrapped up in hemp cloths and in the usual way laid between plates in a hydraulic press and pressed, at first cold, and later on with admission of steam, until the fluid contents are completely exhausted. A solid residue, about one-half of the mass, remains in the press-cloths, while an equal quantity of watery fat runs into the reservoir. This is also reduced to about one-half its weight by the various operations of the refining process, so that the average yield will be about 25 per cent. of salable fuller's fat.

The crude hydrous fat in the press reservoirs yet requires refining and dephlegmation. For the purpose of refining the fat is placed in copper tanks  $3\frac{1}{2}$  feet in diameter and 5 feet deep, which are fixed in iron casings. According to the greater or lesser purity of the fat,  $\frac{1}{5}$  or  $\frac{1}{4}$  of its volume of water and 2 to 3 per cent. of its weight of sulphuric acid of  $66^{\circ}$  B. are added and the whole is heated by direct introduction of steam to moderate boiling, which is kept up for one hour. The steam is then cut off, the mass allowed to settle for a few hours, and the lower turbid and slimy stratum drawn off. The liquid running off is replaced by an equal quantity of pure water, and the whole heated to moderate boiling in order to remove adhering sulphuric acid.



The whole is then allowed to settle for 12 hours, when, after removing the aqueous stratum, the clear mass of fat is drawn off. The fat thus obtained still contains a considerable quantity of water, and is freed from it by bringing it into a kettle provided with a copper or iron coil through which steam circulates.

The fat thus obtained forms a thickly-fluid, oily mass of a brown to black color and disagreeable odor, and is brought into commerce under the name of "fuller's fat." It is much used in the manufacture of soap, though never by itself, but always in combination with other fats, especially with palm-kernel oil and resin. It is much liked for soaps used in the textile industries. It readily saponifies with tank-lye of  $18^{\circ}$  to  $24^{\circ}$  B.; it must, however, be strongly salted out, and is best further boiled upon a second water, whereby it yields a good grain-soap of fair washing power. It shows, however, varying colors, every lot of fat yielding in this respect a different product.

In buying fuller's fat it is well to test it, as it frequently contains much dirt and water, and recently complaints have been made about mineral oil being found in it. The latter is very likely not due to intentional adulteration, but to the fact that at the present time mineral oils are much used in spinning establishments and reach the wash-waters.

Fuller's fat consisting chiefly of fatty acids can be readily distilled with superheated steam. The fat thus refined is commercially known as "solid white olein." It readily saponifies with tank-lye of  $20^{\circ}$  to  $25^{\circ}$  B., and yields a beautiful, firm grain-soap of a silver lustre and a good odor, which is very much liked as a fulling soap. This distilled fat can also be advantageously used in the fabrication of white grain-soap.

*Resin or colophony.*—Resin is obtained from turpentine, a mixture of oil of turpentine and resin. In regions rich in pine forests, especially in France and the United States, the trees are "boxed," *i. e.*, excavations are made into the trunks of the trees about six inches or more above the roots. The crude turpentine exuding is collected in barrels and transferred to stills. If the turpentine is distilled with water, oil of turpentine passes over and a resinous mass remains as a residue which is known as "boiled turpentine." If this is melted without water until it

becomes clear, then the colophony or resin remains. The grade of resin depends first upon the quality of the turpentine, and, second, upon the skill in distilling. "Virgin turpentine," if skillfully distilled, will yield what is known as "window-glass resin," of which there are two or three grades. If by any means water gets into prime resin, it becomes opaque. This accidental addition of water must take place after the resin has been drawn off from the still.

"Yellow-dip turpentine," which is the running of the second and subsequent years from the trees, yields the medium grades of resin, while the "scrapings," *i. e.*, the inspissated gum from the tree facings, yield an inferior resin, from very dark to almost black. All varieties of resin are brittle, of a conchoidal fracture, and a more or less strong odor of oil of turpentine. The specific gravity of colophony is 1.08.

Common resin is soluble in alcohol, ether, and in fat and volatile oils; it melts at  $272^{\circ}$  F., and, when heated to a higher temperature, becomes darker. By exhausting colophony with cold alcohol, sylvic acid ( $C_{20}H_{30}O_2$ ) goes into solution, and on evaporation crystallizes in flat needles, melting at  $264^{\circ}$  F. and solidifying to an amorphous mass. Besides sylvic acid colophony contains the isomeric but amorphous pinic acid. A third isomeride, called pimaric acid, exists in galipot, the resin of *Pinus maritima*.

Besides its application in soap-making common resin finds varied uses. The most important of these are the manufacture of varnishes, lacquers, cements, brewers' and bottlers' pitch, and of lubricants for wagons and machinery.

Though common resin cannot be called an actual fat, it can be readily saponified with strong lyes. Formerly it was much used in combination with tallow and palm-oil for grain-soaps; at the present time it is, however, chiefly employed in combination with palm-kernel oil, cocoanut-oil, bone-fat, olein, fuller's fat, and cotton-seed oil.

A method of fabricating resin grain-soaps, formerly much in use, was to boil the tallow to a clear paste with an average lye of  $13^{\circ}$  to  $15^{\circ}$  B., then salt out, and, after removing the sub-lye, boil the grain clear upon the lye of  $24^{\circ}$  to  $25^{\circ}$  B. required for the

resin. The comminuted resin was then added and saponified over a moderate fire. After further boiling the thoroughly fitted and separated soap was slightly ground with water. Saponification of the resin with strong lyes is necessary, because, with the use of weak lyes, the formation of froth would be promoted by the large amount of moisture naturally brought into the soap by them. A soap from 100 parts of tallow and 60 to 100 parts of resin was also frequently prepared by boiling the tallow saponified with lye of  $15^{\circ}$  B. to a paste quite free from froth, adding the resin together with the corresponding quantity of lye of  $24^{\circ}$  B. and boiling like Eschweg soap. The soap was then slightly fitted and separated with caustic soda of  $36^{\circ}$  to  $38^{\circ}$  B. until strong "wetting" appeared.

At the present time, with the use of palm-kernel oil, etc., where stronger lyes are employed from the start, the preparation of good resin grain-soap is much simpler; care must, however, be had not to add too much grain-paste from former boilings, whereby much injurious salt is frequently introduced into the soaps. The fitting of resin grain-soap must always be done successively and carefully adjusted; and be rather light. A resin grain-soap from 100 parts of palm-kernel oil and about 30 parts of resin is frequently prepared by boiling the palm-kernel oil to a clear paste with lye of  $24^{\circ}$  to  $25^{\circ}$  B. The fire being moderated, the comminuted resin is then added together with the required quantity of lye of  $24^{\circ}$  to  $25^{\circ}$  B. and allowed to thoroughly combine. After the continuing boiling for some time, the soap is strongly fitted, salted out with caustic soda lye of  $38^{\circ}$  B., and ground with water until it thoroughly "wets." For the advantageous use of caustic soda in the preparation of such soaps, dissolve together with 100 pounds of 74 to 76 per cent. caustic soda about 25 to 28 pounds of calcined soda.

According to another method, with the use of half-bone fat and half palm-kernel oil and 25 to 30 per cent. of pale resin, the bone-fat is boiled to grain with tank-lye of  $13^{\circ}$  B. In the mean while the palm-kernel oil and resin are boiled to a clear paste with caustic soda lye of  $20^{\circ}$  to  $23^{\circ}$  B. This paste is then added to the grain from the bone-fat, and the whole, after boiling until free from froth, is allowed to stand for several hours, when



the soap is ladled out. The precipitate remaining in the kettle is then salted out and crutched into the soap in the frames. This method, which yields a solid soap, requires, of course, pure fats and pale resin.

According to the American method, resin soaps from 100 parts fat and 100 parts resin are generally prepared by saponifying tallow and about 10 per cent. of palm-oil with lye of  $13^{\circ}$  B., salting out and boiling the grain clear; after removing the sub-lye, the lye of  $25^{\circ}$  B. necessary for the saponification of the resin is added and then successively the pulverized resin. The whole is then allowed to boil for some time, adjusted to "touch," and again separated. The soap is then thoroughly ground and allowed to rest for some time in the well-covered kettle. The soap is then brought into the frame and a strong solution of crystallized soda (4 to 5 parts by weight to 100 parts by weight of soap) crutched in, whereby the phlegm is absorbed and the soap becomes firmer; and is then crutched cold.

A yellow or reddish-yellow color is generally given to light resin grain-soap by boiling some crude palm-oil with it.

For the fabrication of dark resin grain-soaps, palm-kernel oil, wool fat, fuller's fat, kitchen fat, etc., with an addition of American F resin, are generally used. The fats are first saponified, the pulverized resin and the corresponding quantity of lye of  $24^{\circ}$  B. added, and, after thoroughly fitting and salting out, the grain is boiled clear and then ground with hot water until the sub-lye commences to paste. For marbled soap, the grain is brought into the frame, drawn through with a rod, and well covered. For very dark soaps some diluted sugar color is added, or some pitch boiled with the soap.

In many regions, besides resin grain-soap, light and dark paste soaps with a varying per cent. of resin are manufactured, which are frequently filled with water-glass, talc, clay, etc. These soaps are mostly prepared by boiling cocoanut-oil or palm-kernel oil (for dark soaps with an addition of 5 to 10 per cent. of fuller's fat) with caustic soda lye of  $23^{\circ}$  B.; the addition of resin varies generally between 10 and 20 per cent. The method of boiling is, as a rule, that of an ordinary cocoanut-oil soap. The oil and resin are saponified with the lye, and when the paste is clear the

fire is moderated and a corresponding quantity of potash solution of  $28^{\circ}$  to  $30^{\circ}$  B. added. Combination being established, the mass is shortened with salt water of  $20^{\circ}$  B., and finally 15 to 20 per cent. of heated soda water-glass, into which a few pounds of talc have been stirred, is crutched in. The soap has to be crutched in the frame until quite cold, as otherwise it readily marbles. A too strong fitting has to be avoided, as otherwise the soap would show strong efflorescence, especially in the cold season of the year.

Besides the above-mentioned resin soaps, which are prepared in the warm way, many soap-boilers manufacture them by the cold way. The principal materials used are tallow, palm-oil, cocoanut-oil, and palm-kernel oil; the addition of resin varies between 10 and 100 pounds to 100 pounds of the fat used. The preparation of these soaps is effected in a manner similar to that of cocoanut-oil soap in the cold way. Fat and resin are melted together and then stirred together, at  $122^{\circ}$  to  $189.5^{\circ}$  F., with caustic soda lye, the quantity of which depends on that of the resin used. Combination being established, the soap is brought into the frame and the latter covered.

Resin finds a further and important application in the manufacture of the various smooth and artificial grain soft soaps. The resin—about 5 to 15 pounds to 100 pounds of oil—is either at once brought into the kettle together with the oil and saponified with it, or it is added together with the required lye of  $30^{\circ}$  B. to the soap when boiling up, finished, and combined with it by crutching. The latter method, which is chiefly used and can be particularly recommended for glycerin soft soap and similar varieties, gives paler soaps and a somewhat larger yield, though refitting is necessary after the addition of the resin. On the other hand, by boiling the resin with the fat, the soap is somewhat darker and the yield somewhat less, but refitting being omitted, the work is quicker and surer. By an addition of resin pure soft soaps are rendered cheaper, give a better lather, and become more lustrous; they are, however, considerably softer, and it is, therefore, necessary to use about 20 to 30 per cent. of soda lye of about  $24^{\circ}$  B., the quantity depending on the season of the year; more being used in summer and less in winter.

It is sometimes desirable to have a very light resin. This can

be obtained by artificial bleaching. Melt the resin in a kettle and allow it to stand until all dirt has settled on the bottom. The clear resin is then ladled into another kettle and to each 100 pounds of it are added 20 pounds of common salt solution of 9° B. The whole is then boiled for one hour, when the fire is diminished. As soon as ebullition ceases the resin settles on the bottom, while the salt lye separates as a brownish fluid on the top. This salt lye is drawn off, the salt water renewed, and the whole again boiled. If the resin is not sufficiently decolorized, the operation is repeated for the third time.

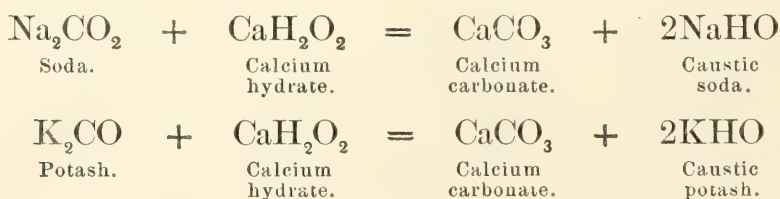


## CHAPTER VI.

## ALKALIES.

By the term "alkalies" are designated the oxides of a small group of metals which are distinguished by being lighter than water, oxidizing readily in the air, and decomposing, at an ordinary temperature, water, with the development of hydrogen gas. The oxides of these metals—the alkalies—are the strongest bases known; they combine with water to hydrates, forming the so-called caustic alkalies. These have a caustic, lye-like taste, destroy the skin and all organic tissues, and are readily soluble in water. Their solutions color reddened litmus tincture blue; the coloring matter of violets and roses, green, and that of turmeric, brown; they show, as it is termed, an alkaline reaction. From the air these hydrates absorb water and carbonic acid.

Two of these alkaline hydrates are extensively used for technical purposes, potassium hydrate (KHO) or caustic potash, and sodium hydrate (NaHO) or caustic soda. The usual method of converting the alkaline carbonates into alkaline hydrates is by means of slaked lime (calcium hydrate  $\text{CaH}_2\text{O}_2$ ). By bringing together solutions of alkaline carbonates with slaked lime a conversion takes place, by the carbonic acid of the alkali combining with the calcium oxide, to calcium carbonate, which, being insoluble in water, falls to the bottom, while the alkali combines with the hydrate of the lime and remains in solution :—



Following hereto we give the manner of manufacture and the properties of the various kinds of alkaline carbonates and caus-

tic alkalies found in commerce, commencing with soda, which, next to common salt, is the most important of all mineral salts.

SODA.—The soda brought into commerce is known, according to its derivation, as (1) Natural soda and (2) Artificial soda.

1. *Natural soda*.—Sodium carbonate is widely distributed in nature as a constituent of many minerals and mineral waters; it further exists in the soda lakes of Hungary, Egypt, and America, and in the water of the geysers of Iceland. It is occasionally found native as an efflorescence on the soil, for instance, on the alkali plains of North America, on the steppes between the Black and Caspian seas, in Mexico, South America, etc.

The oldest-known occurrence of natural soda is in Lower Egypt, where, in the neighborhood of Memphis and Hermopolis, a variety of soda is obtained from some shallow lakes, which is known as “latroni,” and contains besides sodium carbonate, Glauber’s salt, common salt, sand and water. Somewhat different from this is the “trona,” an efflorescence on the soil occurring also in Egypt, which mostly contains sodium sesquicarbonate.

In Columbia a variety of soda known as “urao” crystallizes from a lake during the hot season of the year; it is also a sesquicarbonate.

Extensive deposits\* of the chloride, sulphate, and carbonate of soda are found at many points in the arid regions of the United States, and may be had for the trouble of gathering. These deposits occur in the desiccated beds of ancient lakes in Nevada, Arizona, Western Utah, and portions of California and New Mexico. There are certain lakes, also, which are valuable brines.

In the basins where evaporation has been nearly or quite complete, the alkaline salts occur either at the surface, when they appear like fields of snow, frequently many square miles in extent, or they may be concealed beneath the layers of fine mud known as playa deposits. Again, large areas in Nevada and Arizona are white with alkaline salts that have been brought to the surface in solution and deposited when the waters evaporated. These efflorescences are frequently rich in sodium carbonate, sul-

\* I. C. Russel, in United States Independent Journal, 1886.

phate and borate, and have been utilized to a limited extent at a few localities.

The lakes of the Far West, which are likely to become of commercial value on account of the alkaline salts they contain, are Great Salt Lake, Utah; the Soda lakes near Ragtown, Nevada; Mono and Owen's lakes, California; and Summer and Abert lakes, in Oregon. All of these are without outlet, and owe their high percentage of mineral matter to the concentration by evaporation of the waters of streams and springs with which they are supplied. Their chemical composition is shown in the following table:—

	1	2	3	4	5
	Great Salt Lake, Utah, 1899.	Soda Lake, Nevada, 1883.	Mono Lake, California, 1883.	Owen's Lake, California, 1876.	Abert Lake, Oregon, 1883.
Sodium . . .	49.690	40.919	18.100	21.650	2.773
Potassium . . .	2.407	2.357	1.111	2.751	10.637
Calcium . . .	0.225	—	0.278	trace	—
Magnesium . . .	3.780	0.245	0.125	trace	0.002
Lithium . . .	trace	—	—	trace	—
Chlorine . . .	83.946	40.851	11.610	13.440	8.220
Bromine . . .	trace	—	—	—	—
Carbonic acid . . .	—	16.584	11.465	13.140	4.547
Sulphuric acid . . .	9.858	11.857	6.520	9.362	0.497
Phosphoric acid . . .	—	—	—	trace	—
Nitric acid . . .	—	—	—	trace	—
Boracic acid . . .	trace	2.026	0.153	trace	—
Silica . . .	—	0.278	0.268	0.164	0.064
Alumina . . .	—	—	—	trace	—
Total parts per thousand	149.906	115.117	49.630	60.507	26.740

The analysis of No. 1 was made by Prof. O. D. Allen, of Nos. 2 and 3 by Dr. M. T. Chatard, of No. 4 by Dr. Oscar Loew, and of No. 5 by Dr. T. W. Taylor, all connected with the United States Geological Survey.

It is safe to predict that Great Salt Lake will not only be of great value in the near future on account of the immense quantities of common salt it is capable of producing, but also for the sodium sulphate it contains. When the temperature of the lake-water is reduced to 20° F., the separation of sodium sulphate takes place as a flocculent precipitate, which increases in quantity



with decrease of temperature. This should suggest to manufacturers a method of obtaining the salt and on a large scale. When the temperature of Great Salt Lake is lowered on the approach of winter, its waters become opalescent, owing to the precipitation of sodium sulphate in an extremely finely divided state. During the winter months the temperature of the air in the region of the lake sometimes falls to  $20^{\circ}$  or more below  $0^{\circ}$  F., and at such times the separation of sodium sulphate takes place on an immense scale, and it is thrown upon the shore in thousands of tons. The amount that could be gathered at such times is practically unlimited. As railroads now touch the shore of the lake, the problem of supplying this salt to manufacturers is simplified.

The Soda Lakes, situated on the Carson Desert, Nevada, about fourteen miles east of Wadsworth, have already been utilized as a source of sodium carbonate which is shipped to San Francisco. These lakes occupy the craters of extinct volcanoes, and the mineral matter they contain has been derived mainly from the leaching of the lopilli and lacustral deposits surrounding them.

Mono and Owen's lakes are now quite accessible by rail, and are capable of furnishing immense quantities of sodium sulphate and carbonate. It has been estimated by Dr. Oscar Loew that Owen's Lake contains about twenty-two million tons of sodium carbonate and a little less than one-third of this amount of sodium sulphate. It is estimated that Mono Lake contains—

Potassium chloride . . . . .	8,998,856 tons.
Sodium chloride . . . . .	73,524,285 “
Sodium sulphate . . . . .	40,636,089 “
Sodium carbonate . . . . .	78,649,194 “
<hr/>	
Total of salts in lake . . . . .	201,808,424 “

Summer and Abert lakes, situated in Southern Oregon, are remote from railroads, but are extremely valuable brines on account of the potash salts they contain. These lakes occupy depressions in the bed of an ancient lake of large size, now desiccated, and are very similar in character. Abert Lake alone has been analyzed, but it is probable that its companion has nearly an identical composition. Abert Lake is about fifteen miles long

by five miles broad, and has an average depth (varying with the seasons) of approximately ten feet. Summer Lake is perhaps a third larger and is also shallow, but its average depth is unknown. The percentage of potassium salts in Abert Lake is greater than in any other lake the composition of which has been published, amounting to five-sevenths of the total of solids in solution.

With these abundant resources at hand, the alkali industry of the Far West unquestionably has a great future, and it is to be hoped that it will soon receive the attention that its importance demands.

The occurrence of soda as an efflorescence in the great Hungarian plain was already known to the Romans. The sand of the Theiss plain consists chiefly of minerals containing sodium, which, being disintegrated by the action of lime, water, and air, give rise to a thick crystalline layer of salt which contains 6 to 15 per cent. of sodium carbonate, and is known as "Székso." It is found in greatest abundance in the region between the Danube and the Theiss and in the Debreczine forest from Keeskemét south to Szegedin. By refining this szeksó a product containing 70 to 75 per cent. soda is obtained.

Formerly the principal source of commercial soda was the ashes of plants growing on salt plains, near salt springs, and on the border of the sea. Plants growing in the sea itself are not suitable for this purpose, as they contain but a small quantity of alkaline carbonates, and they are principally worked on account of their content of potassium sulphate and potassium chloride, and especially of iodine. The preparation of soda from the actual soda plants, which are found along the borders of the sea to within a certain stretch into the interior of the country, is very simple, consisting only in the incineration of the plants. The industry was, and is partially at the present time, carried on on the coasts of Scotland and Ireland, but chiefly on the Mediterranean, in Sicily and Sardinia, and on the Spanish coast in the province Valencia, in Morocco and in Armenia, and the South Russian steppes.

The best soda of this kind was the Spanish, and "barilla," the name under which it is known, was given to all vegetable sodas; even the artificial soda was first known in England as "British

barilla." Barilla is also brought into commerce as Alicante, Carthagea, and Malaga soda. It forms a hard and compact mass of a dark ash or gray-blue color, and contains from 25 to 30 per cent. of sodium carbonate. It is hard and difficult to pulverize and has a sharp alkaline taste. It is obtained from plants specially cultivated for the purpose.

Alicante soda is divided into three varieties: *Soude douce*, *melangée*, and *bourde*. The first forms a well-fused ash-like mass with 20 to 25 per cent. of sodium carbonate; the second, a blackish, blistered mass with a sharp fracture; while the third is a poorer quality mixed with particles of coal and containing much common salt and earthy constituents.

In Southern France two varieties of vegetable soda are produced, the "salicor," or Narbonne soda, with 14 to 15 per cent. of carbonate, in the region around Narbonne, from *Salicornia annua*; and "blanquette," or "soude d'aigues-mortes," with only 4 to 10 per cent. of carbonate, from several other varieties of plants.

Vegetable soda, being only caked vegetable ash not purified by lixiviation, contains all the inorganic constituents of the plants. Hence, when treated with water, there always remains a considerable residue of combinations of lime, iron, etc. The portion soluble in water contains, besides sodium carbonate (and potassium carbonate), alkaline sulphates and chlorides.

At the present time the natural soda has been almost everywhere replaced by the artificial product, it being only used in the countries of its production.

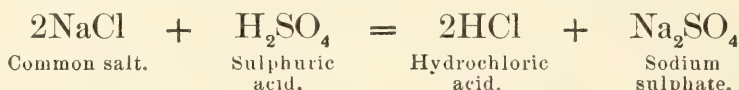
*Artificial soda.*—During the last century the soap industry of France had become so extensive that twenty to thirty millions of francs were annually sent to Spain and other countries for vegetable soda. In 1793, when France, by its war with England, was excluded from intercourse with other nations, and, therefore, dependent on its own resources, the want of potash and soda was much felt in the soap industry. A commission was appointed to examine methods for the fabrication of soda. Of the various processes communicated, the one proposed by Nicolas Leblanc, to manufacture soda from common salt, was considered the only practical one. The industrial process was exposed with such



precision that since that time—nearly a hundred years—very few changes have been made. Leblanc, as previously mentioned, shared the fortune of almost every inventor, so that at the time of his death, in 1806, he was in a state of abject poverty.

Leblanc's method of manufacturing soda consists of heating a mixture of sodium sulphate (Glauber's salt), calcium carbonate, and charcoal, whereby sodium carbonate is formed, with a simultaneous formation of calcium sulphide, caustic lime, and carbonic oxide.

The sodium sulphate is generally prepared from common salt (sodium chloride) by allowing dilute sulphuric acid to act upon the common salt in a suitable furnace at a high temperature; the result is sodium sulphate and hydrochloric acid:—



The calcium carbonate is used either in the form of limestone or chalk. Leblanc recommended charcoal; at the present time hard coal, or sometimes brown coal, is generally used. The fusion of the soda mixture is carried on in reverberatory furnaces, which generally are of a very simple construction, except the mechanical or rotatory furnaces in use in the larger English factories.

The crude soda coming from the furnace consists chiefly of 36 to 40 per cent. of sodium carbonate and varying quantities of calcium sulphide, caustic lime, and calcium carbonate. It also contains small quantities of sodium chloride, sodium sulphate, sodium silicate, various sulphur combinations of the soda with more or less oxygen, etc. In the infancy of the manufacture the crude soda coming from the furnace was directly brought into commerce and bought chiefly by soap-boilers. It was manufactured on a large scale in Marseilles, and as late as 1818 exported in this form to England. But as crude soda on storing in the air is constantly changed by chemical conversions at the expense of quality, it has long ago ceased to be an article of commerce, it being only used in very few places in France. To obtain a purer and more durable product the crude soda is subjected to the process of lixiviation.

To obtain a concentrated solution at as little expense as possible, the lixiviation of the crude soda is systematically carried on in Shanks's apparatus. The crude soda lye thus obtained is concentrated by evaporation and converted into a commercial article by calcination. Evaporation is effected in pans either by bringing heat to bear on the surface of the liquid or conveying it to the bottom of the pan. The salt separated during evaporation is gradually removed by perforated ladles and thrown into a hopper, from which the mother-lye runs back into the pan. When the latter is about half empty fresh lye is run in, and in this way the operation is continued for several months until the mother-lye contaminates the separating salt too much. The mother-lye, which contains much caustic soda and sodium sulphide, is frequently worked into caustic soda; when this is not done, these soda combinations have to be removed from the lye by a process called "carbonization." This is frequently done by evaporating the lye with an addition of saw-dust and calcining. In some large works the calcined salt is re-dissolved, treated with carbonic acid, and again evaporated.

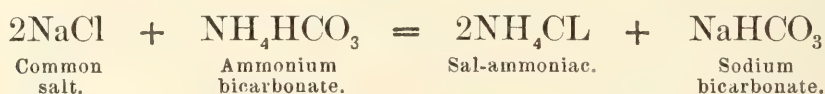
Calcining is effected in a reverberatory furnace, the crystalline carbonate being first drained, which is sometimes performed in centrifugal turbines. By these means a product is obtained containing according to circumstances from 90 to 97 per cent. of sodium carbonate. The soda thus obtained is either ground and then sold, or, for many purposes, again refined. Good calcined soda (second quality) obtained by the described process, should be white but never yellow or reddish; it is, of course, not as white as prime quality. Frequently it shows a bluish color, which may be due to some ultra-marine or sodium manganate, already formed in the crude soda, but in modern times these are sometimes intentionally added. A gray-color of the soda indicates defective carbonization and calcination. Such soda generally contains a considerable quantity of caustic soda and of sulphur combinations.

For many purposes ordinary calcined soda is not sufficiently pure. The caustic soda, and especially the content of iron as well as all that remains as an insoluble residue in dissolving second quality soda, exerts in many cases an injurious influence, especially

where soda has to be used without previous solution as in the manufacture of glass. For the finest qualities of glass and for some other purposes, a refined soda (white alkali) is therefore demanded. The required degree of purity can but seldom be attained by evaporating, draining, etc., of the crude lye, and recourse must therefore be had to calcining and re-dissolving the crude salt. Theoretically refining is a very simple operation, consisting simply of clarifying, evaporating, and calcining. Practically it, however, requires great care and attention to obtain a product answering all commercial demands.

The greater portion of soda is ground before it is brought into commerce. This not only gives a better appearance to the product, but the cost of grinding is much less than the extra expense of packing unground soda which takes up about 50 per cent. more of space.

A plan for the direct conversion of common salt into soda has long been sought. The ammonia process proposed for this purpose is based upon the fact that by mixing a concentrated solution of ammonium bicarbonate with a saturated solution of common salt, sodium bicarbonate will be deposited while sal-ammoniac remains in solution :—



By gradually heating the sodium bicarbonate to redness it loses half of its carbonic acid which can be again utilized, while by treating it with lime or magnesia the ammonia can be regained. The entire reaction is so simple and so readily carried out in the laboratory that it is no wonder many attempts have been made to utilize the process for industrial purposes. H. G. Dyar and J. Hemming, of England, were the first to take out a patent for this purpose; the merit, however, of practically carrying out the process belongs to T. Schloesing and E. Rolland, who, in 1855, established a factory at Puteaux, near Paris, which, however, was abandoned in a short time. The credit of the further development of the ammonia process belongs chiefly to E. Solvay, of Couillet, near Charlevoix, Belgium.

Solvay's process is as follows : A concentrated solution of com-



mon salt is first saturated with ammonia and then with carbonic acid. Ammonium bicarbonate is formed, which, with the common salt, is converted into sodium bicarbonate and sal-ammoniac. The sodium bicarbonate is decomposed, by heating, into sodium carbonate and carbonic acid, the latter being again used for the formation of ammonium bicarbonate. From the solution of sal-ammoniac obtained in the commencement of the process, the ammonia is recovered by heating with lime. The process is, therefore, a continuous one, requiring, independent of the loss of ammonia, only an introduction of common salt and of a portion of the carbonic acid, while soda alone is taken out. Only the lime required for the regeneration of the ammonia and the chloride from the common salt are lost. The entire process seems very simple; it requires, however, very complicated apparatus and a considerable outlay for the establishment of a factory.

Soda prepared by the ammonia process is very pure, being absolutely free from caustic soda, sulphur combinations, and iron. It can be obtained without difficulty so as to show 98 to 99 per cent.

Another variety of soda which deserves mention is that obtained from cryolite, a mineral occurring in Greenland. It consists of fluoride of sodium and aluminium, and when perfectly pure contains in 100 parts 13.07 aluminium, 33.35 sodium, and 53.58 fluor. The soda is obtained by heating the pulverized mineral, mixed with  $1\frac{1}{2}$  times its quantity of chalk, to redness, whereby, under the development of carbonic acid, calcium fluoride and sodium aluminate are formed. The calcined mass is lixiviated; the calcium fluoride remains behind, while the sodium aluminate passes into solution. By the introduction of carbonic acid, generated by the combustion of coal, into the sodium aluminate, it is converted into alumina, which separates, and into sodium carbonate, which remains in solution. The soda solution is concentrated by evaporation, the larger portion of the soda crystallizing out in from eight to ten days. The separated alumina is not pure, but a mixture of 45 per cent. of alumina, 20 per cent. of sodium bicarbonate, and 35 per cent. of water; it is generally further worked for alum.

*Crystallized soda.*—Notwithstanding the great quantity of water (crystallized soda consists of 37.08 parts of sodium carbonate and 62.92 parts of water), for which freight has to be paid, and the further disadvantage that an equal weight of crystallized soda requires far more packing space than calcined soda, it is manufactured on a large scale, not only in soda factories, but in special establishments, chiefly in Northern France and Holland, which buy calcined soda and convert it into crystallized soda. The principal reason why this product can bear the increased cost of freight, packing, and manufacture is due to its purity, of which crystallization is an external evidence. Large quantities of it are used in many branches of industry, even where the calcined article would answer the purpose as well and be cheaper. Its principal consumption is, however, for household purposes, cleansing, washing, etc. For this it is of great importance that it be absolutely free from caustic soda and other combinations attacking the skin. It has the further advantage of being broken up and handled with greater ease than the pulverulent calcined soda, which by exposure to the air cakes together; besides, it is readily soluble in water, while a large portion of the calcined soda remains undissolved as a hard lump in the wash-tub, and pieces of it become mixed with the clothes to their injury.

Though for most purposes a slightly yellow color derived from organic substances would do no harm, an article as colorless as possible is demanded in commerce, and this is but right, it being a guarantee to the consumers that the article is entirely free from iron, etc. Notwithstanding innumerable attempts, it has never yet been found possible to produce salable crystallized soda directly from the crude lyes of soda factories. It is almost exclusively manufactured by dissolving calcined soda with the assistance of heat and allowing the solution to cool in iron vessels. The calcined soda used should be as free as possible from caustic soda and the lower groups of oxidation of sulphur, as well as from sodium sulphide. Coloration due to ferric oxide does no injury, since it remains behind in solution. The calcined soda is dissolved so that the solution shows  $30^{\circ}$  to  $40^{\circ}$  B., the solution is allowed to settle, and the clear lye is run into the crystallizing vessels. The latter are filled nearly to the edge and several

pieces of hoop-iron are laid crosswise over it so as to touch the surface of the liquid. Crystallization begins on this iron frame, a complete incrustation of crystals being soon formed which grows down into the liquid and frequently attains a length of over twelve inches. This incrustation yields the best quality of crystals, those formed on the sides of the vessels, which have to be cut off with a chisel, being less beautiful, while those formed on the bottom are the poorest quality. To avoid a contamination with iron, the vessels have to be kept bright and free from rust; sometimes they are painted.

The mother-lyes always retain some sodium carbonate, and the more the higher the temperature during crystallization. They contain besides all the caustic soda and the greater portion of the sodium chloride and sulphate. These mother-lyes are evaporated in pans to the consistency of paste and then calcined in a reverberatory furnace. They yield a very white soda salt of a weak degree, containing only from 40 to 50 per cent. of pure soda.

Crystallized soda is frequently adulterated with Glauber's salt, and generally for consumers, such as wash-women, to whom the latter is of absolutely no value. According to J. H. Swindells, the manufacture of such spurious crystallized soda is carried on on a large scale, the product being brought into the market as best Scotch soda. The adulteration is, however, readily detected. Dissolve some of the soda, acidulate the solution with hydrochloric acid, and compound with solution of barium chloride; in the presence of Glauber's salt a thick white precipitate is formed.

*Caustic soda.*—Under this name is understood a product consisting chiefly or entirely of sodium hydrate. There is also found in commerce a soda lye which is shipped in glass balloons. For many evident reasons the trade in this article is not very extensive, consumers generally preferring to prepare it themselves from calcined soda and lime. The manufacture of *solid caustic* soda was originally introduced into England, in 1844, by a German named Weissenfeld. The actual commencement of its manufacture on a large scale dates, however, from about 1853, when William Gossage took out a patent, which, besides other improvements in the manufacture of soda, included the gaining of caustic soda from soda lyes by concentration and without the use of lime.



The caustic soda first brought into commerce was colored blue, green, yellow, red, etc., it being only in 1860 that Ralston succeeded in producing the white product by heating the caustic soda to a higher temperature than was formerly customary, *i. e.*, to a temperature at which the iron separates as oxide and the clear caustic soda is left standing over it.

For the manufacture of caustic soda calcined soda was formerly dissolved and made caustic by lime. At the present time this method being too dear has been almost entirely abandoned, the crude lye gained by lixiviating the crude soda being now treated with lime. The lye before being made caustic must be thoroughly clarified and a good quality of lime has to be used. Dilute the crude lye with water to from  $11^{\circ}$  to  $13^{\circ}$  B., as more concentrated solutions cannot be made completely caustic. It is safe to go as high as  $13^{\circ}$  B., and in doing so less water has to be evaporated. Many factories even go as high as  $15^{\circ}$  B., whereby only about 92 per cent. of the soda is made caustic, but a saving in coal is effected. The lye is brought to ebullition and the lime added with constant stirring. It is best to use quick lime, as it is immediately slaked on being added, and the heat developed thereby saves steam. A skilled workman can judge from the manner of boiling, the color of the liquid, and other indications when a sufficient quantity of lime has been added; this can, however, also be tested by filtering a sample and adding some sulphuric or hydrochloric acid, whereby no effervescence must take place. The entire charge can then be either drawn off into a settling vessel, or it is allowed to settle in the pan itself, which requires about half an hour. The clear lye is then gradually drawn off and pumped into vessels for complete clarification. Generally a second operation is carried on in the pan without removing the lime-slime. The pan is again charged with crude lye and water, the contents brought to ebullition and made caustic, somewhat less lime being required than for the first charge, since some caustic lime remains in the slime. After the clear lime is drawn off, the lime-slime is stirred with pure water to a thin paste and brought upon especially constructed filters. The filtrate serves for the dilution of the crude lye. The remaining lime-slime is generally used as a substitute for a portion of the quick lime for

a fresh soda mixture, it being well adapted for this purpose, as it is in a finely divided state and, besides the soda contained in it, is thus utilized.

The caustic soda obtained in this manner is still in a state of great dilution. Its concentration, which is an important task, as it is necessary to save as much fuel as possible, is effected in cast-iron or wrought-iron pans. For the production of a 60 per cent. caustic soda, the lye is evaporated until it shows  $37^{\circ}$  to  $38^{\circ}$  B., and a temperature of  $281^{\circ}$  F. The fire is then withdrawn and the contents of the pan allowed to rest for clarification and the deposit of the salts. For the production of 70 per cent. caustic soda the lye is concentrated in English factories to about  $42^{\circ}$  to  $44^{\circ}$  B. After allowing the contents of the pan to settle, the clear lye is drawn off into clarifying vessels and the salt brought by a perforated ladle into a filter to be returned after draining off to the soda furnace. The clear lye is brought into an iron kettle, generally large enough to hold ten tons of caustic soda, and the fire is started. With a temperature of from  $399^{\circ}$  to  $420^{\circ}$  F., a reddish or sometimes a blackish froth forms on the surface. In most factories manufacturing 60 per cent. caustic soda this froth is removed; it is, however, absolutely necessary to remove it for 70 per cent. caustic soda. For caustic soda of less concentration it is advisable to withdraw the fire when the temperature has reached  $320^{\circ}$  F., so that, by allowing the contents of the pan to clarify once more, more salts may separate and a purer lye be obtained. The boiling is then continued. When the fluid has reached a temperature of  $356^{\circ}$  F., it already contains 53 per cent. of sodium oxide (nearly equal to 70 per cent. of caustic soda) and congeals completely on cooling. It is very dark and of a syrupy consistency, and shows a great tendency to boil over. This is prevented by the workman beating the surface of the fluid in a peculiar manner with a shovel, which separates the froth. When the mass has reached a temperature of  $401^{\circ}$  F. ebullition ceases almost entirely and but little vapor escapes, though the mass still contains nearly 20 per cent. of water. At about  $460^{\circ}$  F. the mass contains almost exactly 60 per cent. of sodium oxide =  $77\frac{1}{2}$  per cent. caustic soda.

At this stage the contents of the pan show scarcely any motion,

there being but a slight ebullition on the edge of the pan. The vapor, which is still developed, carries along small quantities of caustic soda, which produce a very disagreeable, stinging sensation upon the skin. The surface of the mass becomes covered with a lustrous coat of graphite, while a reddish separation of salts is formed around the edge of the kettle. An iron lid is now placed upon the kettle and the fire made as strong as possible. When the mass has reached the proper temperature the complete oxidation of the sodium sulphide still present, as well as that of other sulphides and cyanides, is finished either by the addition of sodium nitrate or by blowing in air. A sample is then taken and examined as to its alkalinity. The color of the mass varies from light brown to deep red. The contents of the kettle are now allowed to clarify in the kettle itself, which generally requires eight to twelve hours, a strong fire being kept up during this time. The quality of the product depends on complete clarification. The mass is then run into sheet-iron drums; if it is not entirely clear and colorless the solid caustic soda will show defects. The sediment in the boiler, which amounts to about 9 to 11 per cent. of the mass, is generally brought into iron boxes and, after cooling, is broken up and again dissolved. The solution is brought to  $28^{\circ}$  B., allowed to settle, and the clear liquid added to the crude lye to be rendered caustic. The residue, which chiefly consists of ferric oxide, is thrown away.

For the production of 76 per cent. caustic soda it is preferable to remove the sodium sulphite from the diluted lyes, instead of waiting to the end of the operation. This is best effected by a suitable metallic oxide. The Greenbank Alkali Works Co., that first introduced caustic soda of a high degree, and furnish at the present time an excellent product, use plumbic oxide (or litharge) for the purpose.

*Commercial valuation of soda.*—The commercial valuation of soda is differently indicated by the three principal countries of its production: in Germany according to per cents. of sodium carbonate, in England according to per cents. of actual or available soda, and in France according to degrees of Descroizilles founded upon an arbitrary basis. The designation according to German degrees seems to be the most rational one for ordinary soda, but



is a very unfortunate one when applied to caustic soda. The English method, according to per cents. of available soda, is decidedly the best. Under available soda is understood everything that acts upon the test acid, because in the manufacture of soap it acts in the same manner as soda. In France these degrees are called Gay-Lussac's degrees; they are, however, never used in practice. Pure sodium carbonate contains, according to the English designation, 58.49 per cent. As sodium oxide is understood as a constituent of the hydrate, it is perfectly correct to apply the same degrees to caustic soda. If an Englishman, therefore, speaks of a soda of 52 per cent., it means that the test acid neutralized by the soda corresponds to a quantity of  $\frac{52}{100}$  of the weight of sodium oxide used, which may, however, be present as carbonate, silicate, aluminate, hydrate, and even as sulphate. Unfortunately, in practice an error has crept into this otherwise rational designation which thus far has only been partially eradicated. The old incorrect equivalent of sodium of 32 is still used instead of 31, and the test acids have been regulated accordingly. Thus too high a percentage is obtained, which does not correspond to the actual percentage of sodium oxide, the value being made to appear higher by  $\frac{1}{31}$  than it actually is. Moreover, there is a further difference between the Tyne and Lancashire manufacturers, the former basing their valuation on the equivalent of sodium carbonate which they take at 54 instead of 53, the test acid being prepared so that 1 litre of it saturates 54 grammes of pure sodium carbonate. Pure sodium carbonate, with this test acid, therefore, indicates  $\frac{32 \times 100}{54} = 59.26$  per cent., instead of 58.49 per cent., and hence 0.77 per cent. too much. Every English statement of degree shows, therefore, 1.316 per cent. too much of its total amount; 50 per cent. of actual sodium oxide show, for instance,  $50 + 50 \times 0.0136 = 50.66$  English degrees, as calculated by the Tyne manufacturers, as well as by the principal commercial analysts in England. In Liverpool, however, a practice has been gradually established which is not even based upon an erroneous equivalent, but simply intended to deceive the consumer. It is there customary to say: "As the 'old' equivalent of the pure sodium carbonate is  $\frac{1}{31}$  larger than the 'new,'

all we need to do is to increase our per cent. figures found according to the actual equivalent by  $\frac{1}{54}$  in order to obtain the commercial valuation; hence, we call 53 per cent. sodium oxide 54 per cent." We would, however, remark that certain large English firms send out their soda ash according to the real equivalent.

The most irrational of all methods of valuation is that according to degrees of Descroizilles, in general use in France. These degrees indicate how many parts of pure mono-hydrated sulphuric acid (double oil of vitriol) are neutralized by 100 parts of the soda ash in question. As the equivalents of sodium carbonate and double oil of vitriol are as 53 : 49, 100 parts of pure sodium carbonate require 92.41 parts of double oil of vitriol, and, hence, show as many degrees of Descroizilles.

To avoid the tedious calculation of one method of valuation into the other, we give a comparative table which shows the actual per cents. of sodium oxide (Gay-Lussac's degrees), of sodium carbonate (according to German and English degrees), and Descroizilles's degrees.

Gay-Lussac's degrees.	German degrees.	English degrees.	Descroizilles's degrees.	Gay-Lussac's degrees.	German degrees.	English degrees.	Descroizilles's degrees.
30	51.29	30.39	47.42	54	92.32	54.71	85.35
31	53.00	31.41	49.00	55	94.03	55.72	86.93
32	54.71	32.42	50.58	56	95.74	56.74	88.52
33	56.42	33.43	52.16	57	97.45	57.75	90.10
34	58.13	34.44	53.74	58	99.16	58.76	91.68
35	59.84	35.46	55.32	59	100.87	59.77	93.26
36	61.55	36.47	56.90	60	102.58	60.79	94.84
37	63.26	37.48	58.48	61	104.30	61.80	96.42
38	64.97	38.50	60.06	62	106.01	62.82	98.00
39	66.68	39.51	61.64	63	107.72	63.83	99.58
40	68.39	40.52	63.22	64	109.43	64.84	101.16
41	70.10	41.54	64.81	65	111.14	65.85	102.74
42	71.81	42.55	66.39	66	112.85	66.87	103.32
43	73.52	43.57	67.97	67	114.56	67.88	105.90
44	75.23	44.58	69.55	68	116.27	68.89	107.48
45	76.94	45.59	71.13	69	117.98	69.91	109.06
46	78.66	46.60	72.71	70	119.69	70.92	110.64
47	80.37	47.62	74.29	71	121.39	71.93	112.23
48	82.07	48.63	75.87	72	123.10	72.95	113.81
49	83.78	49.64	77.45	73	124.81	73.96	115.39
50	85.48	50.66	79.03	74	126.52	74.97	116.97
51	87.19	51.67	80.61	75	128.23	75.99	118.55
52	88.90	52.68	82.19	76	129.94	77.00	120.13
53	90.61	53.70	83.77	77	131.65	78.01	121.71

POTASH.—Commercial potash forms a mixture of salts, the principal constituent of which is potassium carbonate. When calcined it is a hard but light, porous, granular mass with a white color shading into pearl-gray, yellowish, or bluish. Separate pieces frequently show blue or red stains upon the fracture. The red coloration is due to ferric oxide, the gray to admixed particles of coal, and the blue to the formation of a small quantity of potassium manganate by the action of the alkali upon manganic oxide. Potash has a strong alkaline taste and is inodorous. It readily dissolves in water, a considerable quantity of indissoluble constituents frequently remaining behind. It absorbs moisture from the air, deliquescing thereby. Its solution shows an alkaline reaction. It melts at the beginning of a red heat.

Four varieties of potash are found in commerce : 1, from wood ashes ; 2, from the residue of beet-root molasses ; 3, from wool sweat (suint) ; 4, from potassium sulphate according to Leblanc's process.

*Potash from wood-ashes.*—The industrial manufacture of potash from wood-ashes is carried on only in countries where wood is abundant, as in Russia, Illyria, Hungary, and the United States.

The process of manufacture is very simple and may be divided into five operations : 1. Incineration of the plants ; 2. Lixiviation of the ashes ; 3. Evaporation of the lye ; 4. Calcination of the crude potash ; 5. Purification of the potash.

Little need be said about the *incineration* of the plants, the process being not the same in every country. The slower the combustion, however, the more ash is obtained. With a vigorous combustion not only a considerable portion of the ash is carried off by the draught, but a portion of the alkaline salts evaporates on account of the high temperature.

The object of *lixiviating, leaching, or washing the ashes*, is to separate the soluble salts from the insoluble. The former consist chiefly of potassium carbonate, sulphate, and chloride. It is *not* indifferent whether leaching is effected with cold or hot water. Potassium carbonate and chloride dissolve readily in cold water, potassium sulphate, however, with difficulty. By leaching, therefore, with cold water less potash is obtained, but it is richer in



potassium carbonate. But potassium sulphate being also a very valuable salt leaching is generally effected with hot water.

Lixiviation is mostly carried on in wooden vats about  $3\frac{1}{4}$  feet high and  $3\frac{1}{4}$  to 5 feet in diameter. They are provided about 4 to 6 inches above the actual bottom with a perforated bottom, in which is inserted a vertical pipe for the escape of air expelled by the water, which otherwise would have to force its way through the ash. To prevent the carrying away of insoluble constituents of the ash, the perforated bottom is covered with a straw mat, or with a layer of straw several inches thick, over which is generally placed a piece of coarse linen. On one side immediately below the perforated bottom the vat is provided with a discharge-cock. The space between the two bottoms becomes gradually filled with fine particles of ash passing through the straw, and must, therefore, from time to time be cleansed.

The ash is first moistened with water, then brought in small portions into the vat and rammed down. Lixiviation is carried on in a systematic manner, *i. e.*, several vats are placed together forming what is termed "a battery." The lye obtained is of a brown color, due to organic substances extracted from the incompletely carbonized wood by the potassium carbonate. The residue from leaching ashes affords a valuable manure.

The evaporation of the lyes is mostly conducted in shallow cast-iron kettles. Alongside these kettles are placed sheet-iron pans which are heated by the fire under the kettles and serve for preliminary warming. The kettles and pans being filled a strong fire is started. The evaporated liquid is constantly replaced by fresh lye from the pans. When the lye has acquired a syrupy consistency, the supply of fresh lye from the pans is interrupted and the fire moderated. On the side of the kettle a salt crust separates, which becomes thicker and thicker until all the lye is converted into a dry salt-cake, when firing is entirely discontinued. After sufficient cooling the brown, hard salt-cake is broken out by means of a hammer and chisel. This crude product, containing about six per cent. of water, is known in the trade as crude or lump potash. As this method of boiling down and cutting out the crude potash must evidently cause considerable damage to the iron pans, the operation is, in some instances,

conducted in a somewhat different manner. The liquid is stirred with iron rakes, and the salt, instead of forming a hard solid mass, is obtained as a granular powder containing upwards of 12 per cent. of water.

A peculiar kind of potash, consisting chiefly of caustic potash with varying quantities of potassium carbonate, is produced in this country, and brought into commerce under the name of "Red American Potash" or "stone ash." For its production the crude potash liquor is heated to ebullition, and a quantity of milk of lime added according to the intended degree of causticity. The carbonate of lime formed is allowed to settle, when the clear lye is drawn off and evaporated until no more water escapes. It is finally heated nearly to redness in the evaporating pan itself, or in a special pan of thick cast iron, in order to destroy the organic substances and to melt the potash. The liquid potash is then ladled out with a sheet-iron ladle into cast-iron boxes to congeal to a stone-hard mass permeated with bubbles. The product, after removal from the boxes, is broken up and immediately packed into tight barrels. It is always contaminated with ferric oxide and has a dirty red or brown color.

The object of *calcining* the crude potash, which is effected in a reverberatory furnace, is to remove the last particles of water, and specially to destroy the organic substances to which is due the brown color.

Potash obtained from wood-ashes is, as previously mentioned, chiefly a mixture of potassium carbonate, sulphate, and chloride. For some applications of potash these three are almost of equal value, as, for instance, for the fabrication of alum; for most purposes, however, the content of potassium carbonate is the valuable portion. Whether the article is poor or rich in the latter can be recognized by the behavior of potash on exposure to the air in an open vessel. Potassium carbonate is a very deliquescent salt, absorbing moisture from the air with avidity; if, therefore, potash rapidly becomes moist on exposure to air, it is an indication of it being rich in potassium carbonate.

*Potash from the carbonized residue of beet-root molasses.*—For many years wood-ash was the only source of potash. But, of late years, the manufacture of potash salts from the residue or

vinasse left after the distillation of fermented beet-root molasses has become a new branch of industry.

The residue, in case it contains free acid, is first neutralized by an addition of calcium carbonate. It is then evaporated to dryness and the residue heated until carbonization of the organic substances is effected.

The composition of the carbonized residue varies, but may be gleaned from the following approximate analysis :—

Potassium carbonate	.	.	.	.	30 to 35 per cent.
Sodium carbonate	.	.	.	.	18 to 20 “
Potassium chloride	.	.	.	.	18 to 22 “
Potassium sulphate	.	.	.	.	6 to 8 “
Insoluble matter	.	.	.	.	28 to 15 “

In the insoluble matter are contained coal, calcium carbonate, and phosphate.

While the carbonized residue is mostly manufactured in the molasses distilleries it is generally refined in chemical factories. The process is very simple, it being based upon the different proportions of solubility of the various salts. In many factories the carbonized residue was formerly simply lixiviated with water, and the lye thus obtained evaporated to dryness, 100 pounds of carbonized residue yielding by this process 45 to 60 pounds of potash, which showed 50 to 60 alkalimetric degrees. Much of this kind of potash was brought into commerce during the Crimean war, when there was a want of Russian potash, and contributed much to the disfavor with which the article was considered by soap-boilers.

In more modern times a separation of the separate salts is generally effected, whereby a potash is obtained containing still 8 to 15 per cent. of sodium carbonate, which, however, can be reduced to 4 per cent. by redissolving and re-evaporating.

*Potash from wool sweat (suint).*—The wool of sheep is saturated with a yellow, fatty substance, the so-called sweat or suint, secreted by the skin of the animal. It consists of a combination of potassium with the nitrogenous acid of a special fat,\* which is partly in a free state and partly combined with earths to an insoluble

\* Compare wool fat, p. 123.



soap, and of small quantities of potassium carbonate and acetate, alkaline chlorides, and an odoriferous substance. Its content of sodium is very small.

In order to reduce the consumption of fuel to a minimum it is necessary in working suint to obtain as concentrated liquids as possible. For this purpose the wool is pressed into vats and lixiviated with water in such a manner that all the water passes through several lots of wool, and several times through each lot, which is finally washed with fresh water. The solutions are then evaporated to dryness, and the residue heated to a red heat in iron retorts, the result being the formation of carburetted hydrogen gas and ammonia, which having been eliminated, the gas is used for illuminating purposes. The carbonized residue in the retort contains the alkaline salts which are recovered by lixiviation with water. The lye thus obtained contains potassium carbonate, chloride, and sulphate, which are separated from each other by evaporating the solution and subsequent crystallization.

*Potash from potassium sulphate.*—While the previously mentioned sources of potash are limited, its manufacture from potassium sulphate, introduced of late years, promises to become a branch of industry of unlimited extent. Much of the potash salts, which find their way into the English market, are derived from the so-called “Strassfurt salts,” produced from the alkaline minerals found in enormous quantities in the valley of the Bode about twenty-five miles southwest of Magdeburg. The manufacture of potash from potassium sulphate was introduced into Germany, in 1861, by Vorster & Grüneberg, of Kalk, near Cologne. The manufacture of potassium chloride is based on the decomposition of carnallite contained in the raw material, in a hot saturated solution, potassium chloride crystallizing out, and magnesium chloride remaining in solution. The hot solution is brought to  $36\frac{1}{2}^{\circ}$  B., diluted to  $35^{\circ}$  B., run into settling tanks, and allowed to crystallize; these crystals, once refined, are almost pure potassium chloride. The next stages of the process are almost identical with those of the Leblanc soda process, the raw material (potassium sulphate) being obtained either from potassium chloride and sulphuric acid by the ordinary sulphate process, or by decomposing the former with magnesium sulphate or

kieserite. Equal weights of potassium sulphate and finely divided limestone or chalk, together with varying quantities of small coal, are roasted together in a reverberatory furnace, the product being an exceedingly impure potassium carbonate. When the decomposition is complete, the molten mass is raked out, broken up when cool, and lixiviated in tanks. The soluble potassium and soda salts are thereby dissolved out, evaporated, and calcined in a small reverberatory furnace.

A carbonate of better quality is produced by following more closely the carbonating operation of the soda process. The liquors from the tanks are evaporated, the potassium chloride and sulphate, which separate out during the concentration, being skimmed off and sawdust is thrown in. The dried salts are then removed to the carbonator and exposed, at first, to a gentle heat, which is, however, finally urged to redness. By this process the sulphur compounds are oxidized into sulphate, and the caustic potash is converted into carbonate. The chief object of the sawdust is to keep the mass of salt open.

*Caustic potash.*—This article has been for a number of years brought into commerce by the Greenbank Alkali Works Co. of St. Helens, England. It has, however, been but little used by soap-boilers, since the lyes prepared from it come too high (about 25 per cent. higher than tank lyes); it is sometimes used to increase the strength of tank lyes. The chemical factory of Buckau, near Magdeburg, and others, having recently commenced the manufacture of caustic potash, a reduction in the price may be looked for, which may enable the soap-boiler to use the article in the manufacture of soft-soaps.

## CHAPTER VII.

## TESTING OF SODA AND POTASH.

THE value of the different varieties of soda and potash depends on their content of pure, effective substance, *i. e.*, of alkaline carbonate or caustic alkali. From the preceding chapter it will be readily understood that the quantity of effective substance varies very much in the alkalies found in commerce, and that the value of a particular kind of soda or potash can, therefore, be ascertained only by determining its content of pure alkali.

For the determination of the value of calcined or crystallized soda, it is sufficient to ascertain the sodium carbonate by the alkalimetric method. It is, however, different with caustic soda, where it is above all required to know how much caustic soda it contains. By directly testing caustic soda by the alkalimetric method an erroneous result is obtained, since the sodium carbonate contained in it is also determined as caustic soda. To avoid this error the sodium carbonate has to be separated previously to titration, which is readily effected by means of barium chloride. By adding to caustic soda solution barium chloride solution a white precipitate of barium carbonate or sulphate is formed in the presence of sodium carbonate or sulphate, while no effect whatever is produced upon the caustic soda. For the examination of soda in this manner, dissolve a weighed quantity of it in hot water and add barium chloride solution until no more precipitate is formed. When all the precipitate has settled on the bottom, filter and rinse with hot water until the water running from the funnel shows no alkaline reaction. The caustic soda in the filtrate is then determined by the alkalimetric method.

With potash a correct result is never obtained by a mere alkalimetric determination, since the sodium carbonate, which is never wanting even in the best potash, is then also calculated as potas-



sium carbonate. The error resulting in this manner is the more serious as sodium carbonate has a lower atomic weight than potassium carbonate and consequently requires a larger quantity of acid for saturation. If, therefore, with a potash containing soda, we calculate the found alkalimetric degrees as potassium carbonate, we obtain a larger sum total than the quantity of the two alkaline carbonates together amounts to. A potash, for instance, which contains 85 per cent. potassium carbonate and 5 per cent. sodium carbonate shows an alkalimetric content of 91.62 per cent., and a potash with 85 per cent. potassium carbonate and 8.6 per cent. sodium carbonate, one of 96.22 per cent. A thorough analysis is, therefore, required for the determination of the value of potash, and as this requires considerable experience in such work, it is best to employ a skilled chemist.

*Alkalimetry.*—The testing of soda and potash by the alkalimetric method is such a simple process that it can be readily learned without much preliminary knowledge of chemistry. To make the processes taking place in the execution of the test clear to persons without chemical knowledge it will be necessary to mention a few chemical laws.

It is a fact determined by innumerable experiments and confirmed by daily experience, that by two bodies acting upon each other, a third body, or, as it is called, a chemical compound, is formed which exhibits properties entirely different from those of either of its constituents, and the proportions of weight of these constituents which form that particular compound admit of no variation whatever. By long-continued trituration, for instance, of mercury and sulphur, cinnabar is formed, the two bodies, however, combining only in the proportion of 200 parts by weight of mercury and 32 parts by weight of sulphur. Every excess of mercury or sulphur above this proportion fails to form cinnabar, the excess of mercury or sulphur, as the case may be, remaining behind. In the same manner 23 parts by weight of sodium combine with 35.5 parts by weight of chlorine to common salt. The numbers which express the proportions of weight in which bodies form combinations are called atomic weights. By careful experiments chemists have determined the atomic weight of every body and have found that, for instance, the atomic weight of

sodium is 23, that of potassium 39, that of calcium 40, that of sulphur 32, that of chlorine 35.5, that of carbon 12, that of oxygen 16, that of hydrogen 1, etc. If, therefore, two or three of the mentioned bodies form a combination, it is done in the proportions of the numbers given. To this must, however, be added that the bodies may also combine in the proportions of twice, three times, or four times the above numbers. Water, for instance, is a combination consisting of  $2 \times 1$  part by weight of hydrogen and 16 parts by weight of oxygen. Soda or sodium carbonate consists of three bodies, viz.,  $2 \times 23$  parts by weight of sodium, 12 parts by weight of carbon, and  $3 \times 16$  parts of oxygen. Hence the chemical law: *When two or more bodies combine chemically, it is done in the proportion of the atomic weights or their simple multiples.*

This most important of all chemical laws finds application in the determination of the constituents of various bodies. The branch of chemistry which occupies itself with the determination of the constituents of bodies is called "chemical analysis." There are two methods of determining the quantity of a body in a given substance, viz., gravimetric analysis, or analysis by weight, and volumetric analysis, or analysis by measure or process of titration (analysis by means of standard solutions). By the first method the substance to be examined is reduced to forms or combinations which are most accurately known as regards the proportions of the quantity of their constituents and admit at the same time of a sharp determination of weight. By volumetric analysis, on the other hand, the quantity of a body is found by reducing it from a determined condition into another also determined condition, by the assistance of a liquid of known effective value and under conditions allowing of a plain recognition of the termination of the reduction. In alkalimetry this final point is recognized by a change of color.

For volumetric determinations a few implements are required which shall be briefly described as follows:—

For the preparation of the *test liquids* or *normal solutions* required for volumetric analysis, serves a flask, Fig. 21, which, up to a mark on the neck, holds exactly 1 liter or 1000 cubic centimetres, and can be closed with a glass stopper. There are also

flasks of the same shape with a capacity of 500, 200, and 100 cubic centimetres. For measuring off small quantities of liquids

Fig. 21.

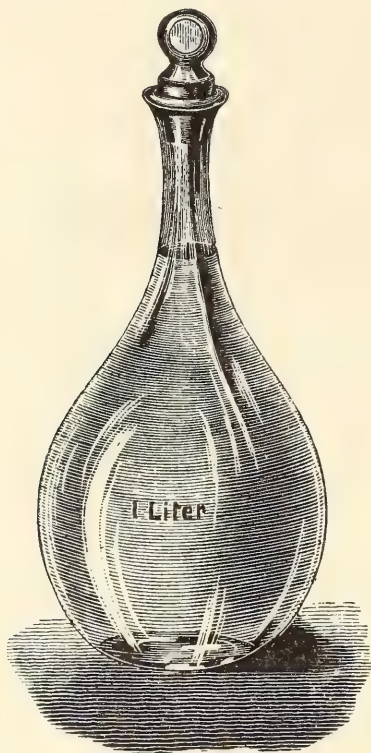
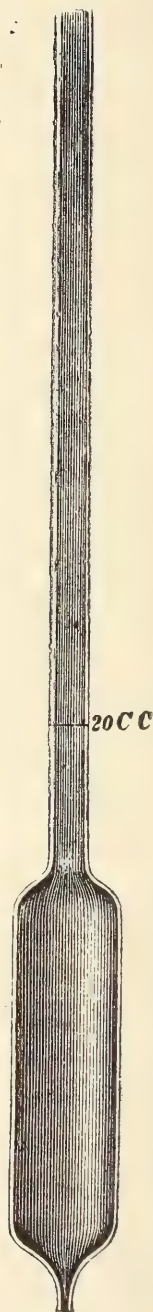


Fig. 22.



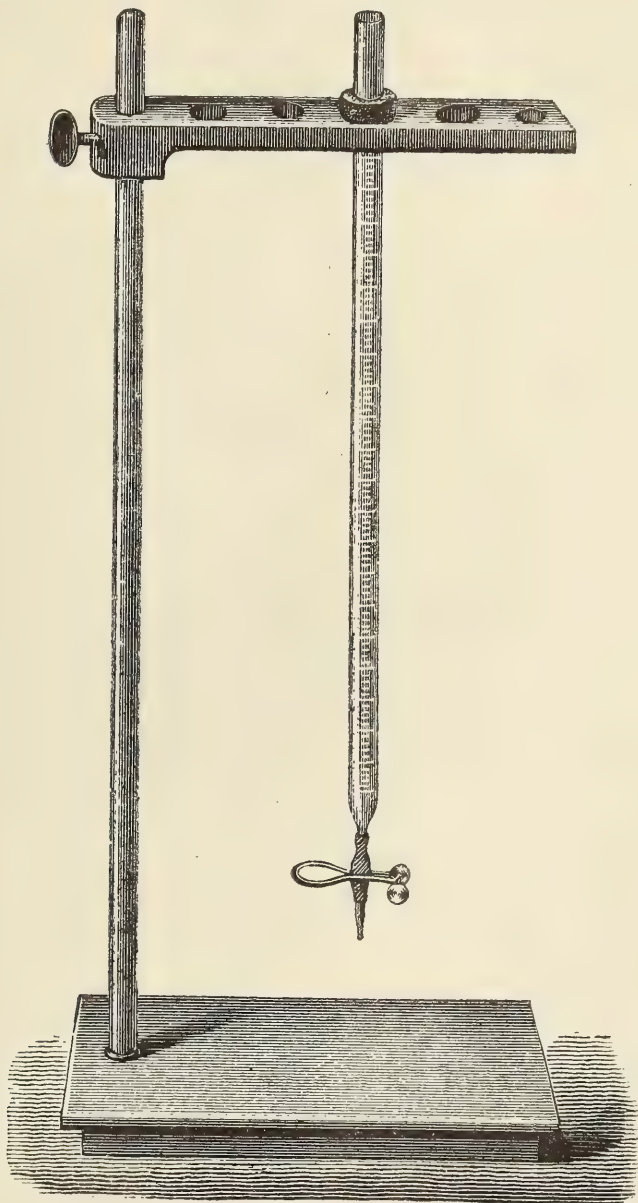
serve a burette and pipette, the latter a glass tube of the form as shown in Fig. 22. It is filled by dipping the lower end into the liquid and sucking on the upper with the mouth until the liquid has ascended nearly to the top. The upper end is then quickly closed with the index finger of the right hand. By slightly lifting the finger, the liquid is then allowed to flow off by drops until its level has reached a mark above the convex expansion, when it will contain exactly the number of cubic centimetres indicated opposite to the mark. There are pipettes of 50, 20, 10, 5, and 1 cubic centimetres capacity.

The burette is a cylindrical glass tube open on the top, graduated, commencing from the top, into whole, one-tenth and one-



fifth cubic centimetres. The lower end of the tube is drawn out to a somewhat distended point so as to allow a rubber tube to be drawn over it and securely fastened. In the lower end a glass tube drawn out to a fine point is inserted. The rubber tube is compressed in the centre by a clip or compression stop-cock,

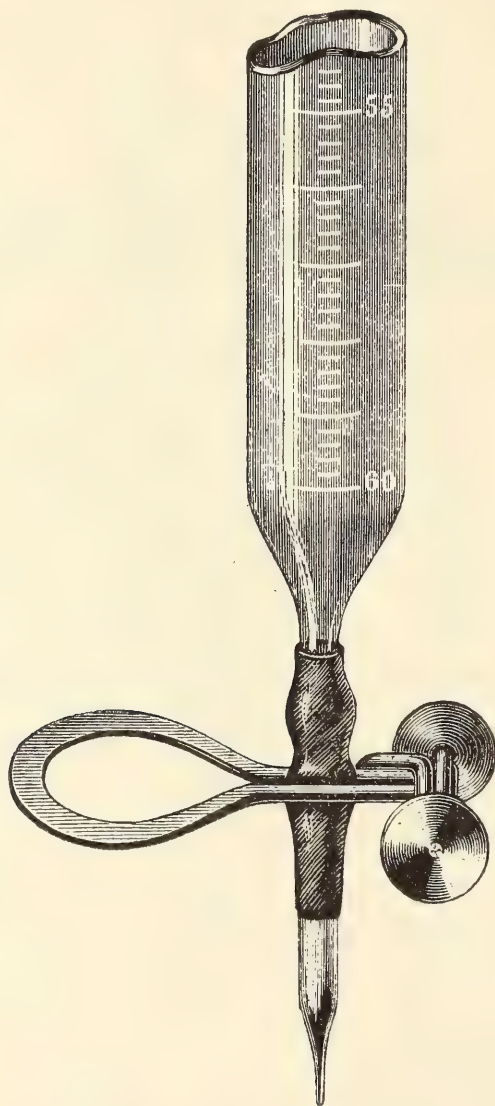
Fig. 23.



whereby the lower end is closed. Fig. 23 shows a burette secured in a stand, while Fig. 24 shows the lower part with the

clip on a larger scale. The burette is filled with liquid from above by means of a small funnel. By a quick, strong pressure

Fig. 24.



upon the handle-joint of the clip, some liquid is then allowed to flow in a jet into a vessel. By this the tube below the clip is filled with liquid and the air contained in it expelled. By a slight or stronger pressure the liquid can, after some experience, be ejected in drops or in a stronger jet. The number of cubic centimetres which have been allowed to flow out can be readily

read off by keeping the surface of the liquid in the tube on a level with the eye.

The determination of effective substance in the alkalies executed with the assistance of the above instruments is based upon the following consideration: Caustic soda lye is a solution of caustic soda in water. By mixing some caustic soda lye with (dilute) sulphuric acid a chemical combination takes place, the result being sodium sulphate (Glauber's salt). Caustic soda and sulphuric acid combine, however, only in determined proportions of weight, namely, 40 parts by weight of pure caustic soda combine with 49 parts by weight of concentrated sulphuric acid. Every excess of caustic soda or sulphuric acid above this proportion remains caustic soda or sulphuric acid and is not converted into Glauber's salt. Now caustic soda, sulphuric acid, and Glauber's salt are representatives of three classes of chemical combinations known as bases or alkalies, acids and salts. These three combinations can be distinguished from each other by the coloring substance known as litmus, a solution of which in water is termed litmus tincture. By coloring water blue with litmus tincture and adding one drop of sulphuric acid, the liquid turns red; it, however, becomes again blue by adding caustic soda lye. But salts, such as Glauber's salt, do not change the color of either blue or red litmus tincture.

This action of the alkalies and acids upon litmus is utilized for our purposes. When to caustic soda solution sufficient litmus tincture be added to perceptibly color the liquid blue, and to this blue liquid, carefully and with constant stirring, sulphuric acid until it just acquires a red coloration, then exactly that quantity of sulphuric acid will have been added which, with the caustic soda present, forms Glauber's salt, *i. e.*, caustic soda and sulphuric acid have been mixed in the proportion of 40 : 49 parts by weight.

Suppose, now, we had 1 liter of dilute sulphuric acid containing exactly 49 grammes of concentrated sulphuric acid and 1 liter of caustic soda lye, containing exactly 40 grammes of caustic soda (these solutions are known as normal acid solution and normal alkali solution), by pouring these two liquids together a liquid would be formed, which contains neither sulphuric acid nor caustic soda, but Glauber's salt, and as a proof that neither



one of them is present, the color of neither red nor blue litmus tincture would be changed by an addition of any of the liquid. Moreover, the quantities of acid and alkali contained in 100, 10, or 1 cubic centimetres of the two solutions would, in the same manner, be just sufficient for the formation of Glauber's salt. To test whether a substance is an acid or an alkali or a salt, litmus paper is suitably used in place of litmus tincture. The solutions of salts, at least those of interest here, are so constituted as not to change the color of blue or red litmus paper; they show a neutral reaction.

From the above it follows that by adding to a large quantity of caustic soda solution 1 liter of dilute sulphuric acid containing 49 grammes of concentrated sulphuric acid, this acid combines with 40 grammes of caustic soda to Glauber's salt, and, further, that by adding only  $\frac{1}{10}$ ,  $\frac{1}{100}$ , or  $\frac{1}{1000}$  liter, or 100, 10, or 1 cubic centimetres of acid,  $\frac{1}{10}$ ,  $\frac{1}{100}$ , or  $\frac{1}{1000}$  part of 40 grammes of caustic soda is converted into Glauber's salt. By adding, therefore, to a solution of caustic soda of unknown content of the above normal sulphuric acid until the added litmus tincture just turns red, the content of caustic soda can be determined by the quantity of sulphuric acid used. If, for instance, 12.5 cubic centimetres of normal sulphuric acid had been used, and as 1 cubic centimetre of acid indicates  $\frac{4.0}{1000}$  gramme or 0.040 gramme of caustic soda, the liquid would contain  $0.040 \times 12.5$  grammes = 0.5 gramme of caustic soda.

The preparation of the normal sulphuric acid would offer no difficulty if the so-called concentrated sulphuric acid found in commerce were pure sulphuric acid. It contains, however, always some water, though the quantity is so trifling that for our purposes, where the highest degree of exactness is not demanded, it may be considered as sufficiently pure. In every drug-store can be found sulphuric acid of nearly 1.842 specific gravity, which is the specific gravity of pure acid. If, however, it cannot be obtained of this strength, it can be readily brought to the commercial standard by carefully adding fuming or Nordhausen sulphuric acid.

Weigh off of this acid, as accurately as possible, 49 grammes, pour it into the liter flask (Fig. 21), rinse the glass in which it

has been weighed several times with water, and pour the latter into the liter flask. Then add sufficient water to fill the flask  $\frac{1}{4}$  full and shake. As by mixing the water and sulphuric acid the liquid becomes heated, allow it to stand for about one hour, and then fill the flask with water exactly to the mark on the neck. After shaking, close the flask with a glass stopper and preserve it for use.

As the normal acid solution prepared in the above manner is not exactly standard, and an error (at the utmost of  $\frac{1}{4}$  per cent.) may be made in the determination of soda, potash, etc., oxalic acid is used for the preparation of the normal acid solution in place of sulphuric acid, where the highest degree of accuracy is required. Pure oxalic acid can be readily prepared from the commercial article by dissolving  $\frac{1}{2}$  pound in as little hot water as possible, filtering the hot solution and allowing it to stand quietly in a cool place. In twenty-four hours the oxalic acid will have separated in needle crystals, which are taken from the water and spread out upon blotting-paper to dry, when they are ready for use.

Solution of oxalic acid behaves towards alkalies in the same manner as sulphuric acid, 40 grammes of caustic soda combining with 63 grammes of crystallized oxalic acid. Therefore, to prepare the solution, weigh off 63 grammes of crystallized oxalic acid, bring it into the liter flask and pour about  $\frac{3}{4}$  liter of water over it. After effecting the solution of the oxalic acid by shaking, fill the flask up to the mark on the neck with water and close it with a glass stopper.

Besides normal acid there is required a normal caustic soda solution containing 40 grammes of caustic soda in the liter. The preparation of this solution is somewhat more difficult, since the solid caustic soda of the shops is by no means pure, containing varying quantities of water. It is, therefore, necessary to choose a roundabout way in order to obtain a caustic soda solution with an accurately determined content of caustic soda. For this purpose dilute about 300 cubic centimetres of pure caustic soda lye with about 500 cubic centimetres of water. Of this liquid measure off into a glass, by means of one of the pipettes previously described, 10 centimetres, color with blue litmus tincture, and

dilute with about 100 cubic centimetres of water. Then add, drop by drop, by means of the previously described clip burette, and with constant stirring, normal acid, until the liquid acquires a distinct red color, which indicates that exactly the quantity of acid required for the neutralization of the caustic soda present has been added. The cubic centimetres of acid used are read off on the burette. Now as 1 cubic centimetre of acid saturates exactly 0.040 gramme of caustic soda, the quantity of caustic soda contained in the 10 cubic centimetres of caustic soda solution can be readily found from the quantity of acid used by multiplying the number of cubic centimetres used by 0.040. Suppose 15.5 cubic centimetres of acid had been used, 10 cubic centimetres of the above caustic soda solution would contain as much caustic soda as is required for the saturation of the acid in the 15.5 cubic centimetres of normal acid. Now, in order to prepare from this caustic soda solution a solution sufficiently diluted so as to become saturated cubic centimetre for cubic centimetre with the normal acid solution, 10 cubic centimetres of it must be sufficiently diluted to make 15.5 cubic centimetres, or as,  $10 : 15.5 = 645 : 1000$ , 645 cubic centimetres of soda lye have to be diluted to 1000 cubic centimetres. These 645 cubic centimetres are then measured off into the liter flask, and after adding water up to the mark on the neck, the flask is tightly stoppered and the contents are shaken.

After these general explanations we proceed to give the manner of testing the alkalies in regard to their effective substance, and, therefore, their real value to the soap-manufacturer, by means of the above-normal solutions.

For the determination of the effective substance in the alkalies, a determined quantity has to be weighed off; for soda and potash it is best done in the following manner: Cut out two square pieces of smooth writing paper of exactly the same size, and place one upon each dish of a balance. Upon one paper place the weight and upon the other bring the substance to be weighed in small pieces until the equilibrium is restored. Caustic soda and caustic potash can be weighed in the same manner, but they being very hygroscopic, which augments their weight, it must be done in a dry-room, and as quickly as possible. It is best, how-



ever, to weigh them inclosed in small glass-tubes tightly stoppered.

Next dissolve the quantity weighed off in about 100 to 150 cubic centimetres of water, color the solution blue with some litmus tincture, then add by means of the clip burette normal acid until the liquid acquires a distinct red color, and read off the quantity of acid used.

For the examination of caustic soda it is best to weigh off 4 grammes and proceed as above. As each cubic centimetre of normal acid used indicates 0.04 gramme of caustic soda in the substance under examination, each cubic centimetre used, calculated to per cents., indicates 1 per cent. of caustic soda.

To draw a simple conclusion as regards the per cent. content of a caustic potash from the number of cubic centimetres of acid used, it is best to weigh off 5.6 grammes, each cubic centimetre used indicating then 1 per cent. of caustic potash. For the determination of caustic soda or caustic potash in a soda lye, weigh off of the former in a small glass 0.4 gramme and of the latter 5.6 grammes, pour the lye into a larger glass, dilute with 100 to 150 cubic centimetres of water, add litmus tincture and then normal acid until the liquid acquires a distinct red color. For each cubic centimetre of normal acid 1 per cent. of effective substance is then to be allowed for in the calculation.

Namely, as 40 grammes of caustic soda and 56 grammes of caustic potash combine with 49 grammes of concentrated sulphuric acid and 63 grammes of oxalic acid, each cubic centimetre of normal acid indicates 0.04 gramme of caustic soda and 0.056 gramme of caustic potash; 100 cubic centimetres, therefore, respectively 4.0 and 5.6 grammes. If, now, in 4.0 grammes of solution or solid substance used are 4.0 grammes of caustic soda, it would indicate a pure substance or 100 per cent. Hence it follows that if, for example, only 15 or 65 cubic centimetres of normal acid are used, only 15, or 65 per cent. of pure substance is contained in the sample under examination, *i. e.*, the number of cubic centimetres used gives at once the content in per cents.

For the determination of the value of soda or potash the process varies somewhat from the above. By adding to a solution

of alkaline carbonate litmus tincture, the liquid acquires a blue color. By adding acid, carbonic acid escapes, which, however, remains partly dissolved in the liquid and colors the litmus tincture red. The quantity of acid required for the decomposition of the alkaline carbonate can, therefore, not be recognized by the change of color. The object is, however, attained in the following manner: Add sufficient acid, so that the liquid shows a strong red coloration, and boil, whereby the carbonic acid is entirely expelled. If after a few minutes' boiling the liquid still remains red, an excess of acid has been added. Now the normal soda solution, prepared as previously described, is so constituted that 1 cubic centimetre of it exactly neutralizes 1 cubic centimetre of acid; by adding, therefore, to the liquid freed from carbonic acid by boiling, normal soda solution until the red color just passes into blue, exactly as many cubic centimetres of it will have been used as cubic centimetres of acid had been added in excess. By deducting these cubic centimetres of normal soda solution from the cubic centimetres of normal acid originally used, the exact quantity of normal acid required for the saturation of the alkaline carbonates is obtained.

As 53 grammes of sodium carbonate combine with 49 grammes of concentrated sulphuric acid or 63 grammes of oxalic acid, it is best to weigh off 5.3 grammes of soda, place them in a small flask of about 200 cubic centimetres' capacity, pour in water, and then heat. When the soda is dissolved, color blue with litmus tincture and add, by means of the clip burette, normal acid until the liquid acquires a strong red color. After continuing the boiling for a few minutes, the liquid should still show a strong red coloration. Then add, by means of the clip burette, normal soda solution, drop by drop, until the liquid, after shaking, just acquires a blue color. Deduct the quantity of normal soda solution used from the quantity of normal acid solution added, the number of cubic centimetres obtained giving the content of pure sodium carbonate in per cents.

For the examination of potash by volumetric analysis, weigh off 6.9 grammes, since 69 grammes of potassium carbonate are neutralized with 49 grammes of concentrated sulphuric acid, and proceed in the same manner as given for soda. Each cubic centi-

metre of normal acid used for neutralization indicates 1 per cent. of potassium carbonate.

To those not having a balance for accurately weighing 4, 5.3, and 6.9 grammes, it is recommended to weigh off larger quantities, namely, 40 grammes of caustic soda, 53 of calcined or crystallized soda, and 69 of potash, and dissolve in so much water that the solution makes exactly 1 litre. For the execution of the test take of this solution, by means of a pipette, exactly 100 cubic centimetres, when each cubic centimetre of normal acid used will correspond to 1 per cent. of caustic soda or sodium carbonate or potassium carbonate.

Normal solutions, already prepared, can generally be had in almost all large drug stores at moderate prices.

Of the normal acid solutions—

1 cubic centimetre =	0.047 grammes of	potassium.
= 0.056	“	caustic potash.
= 0.069	“	potassium carbonate.
= 0.031	“	sodium.
= 0.040	“	caustic soda.
= 0.053	“	sodium carbonate.

Of the normal alkaline solutions—

1 cubic centimetre =	0.064 grammes of	oxalic acid.
= 0.040	“	sulphuric acid.
= 0.049	“	concentrated sulphuric acid.

*Manner of taking samples for analysis.*—In examining a commercial article it is of great importance to obtain an average sample, and for this reason a few hints will here be given in regard to the correct manner of taking samples for chemical analysis.

For the examination of an average sample from several barrels or drums, take from every barrel or drum a sample weighing about three-quarters to one pound and put it in a covered barrel. The covering of the barrel is necessary, as otherwise the original composition of the samples might undergo a change by the evaporation of moisture.

After taking a sample from all the barrels or drums, turn the barrel containing them over on a level, hard, clean surface and mix the mass by spreading it out uniformly in a shallow layer



and reforming it into a pile in the centre, carefully repeating the operation several times. From the mass finally obtained an average sample weighing about four pounds is made up by combining small portions from every part of the spread-out mass. This sample is then again repeatedly mixed in the same manner as the original large sample and then placed in several bottles tightly corked and sealed. The distribution into several bottles is recommended in order to have evidence in case of dispute. For actual use the samples must be further pulverized and mixed.

The taking of samples and all operations connected with it must, of course, be executed as quickly as possible and without interruption, to avoid the danger of the sample undergoing a change by the absorption or evaporation of moisture.

With substances consisting of a mixture of large and small pieces, care must be had in taking the first average sample to preserve the same proportions as in the original. After mixing the various samples, the large pieces are comminuted, the whole is then again mixed, and a small sample is finally taken from every portion of the spread-out mass, which is pulverized and preserved in the manner previously described.

Fig. 25.



Where there is a large number of barrels, so that the taking of a sample from each barrel would be tedious and time-consuming, a sample is taken from every third, fourth, fifth, or tenth barrel, according to the number, and the samples mixed in the previously described manner. For taking samples from barrels it is best to use the instrument shown in Fig. 25. After boring by means of a centre-bit a hole about twelve inches wide in the head of the barrel, the instrument is forced with a screw-like motion as far as possible into the barrel, whereby the concavity becomes filled with portions of the various layers of the contents. The instrument is then withdrawn

without turning. It should be always kept bright and free from rust.

Sample taking, as previously mentioned, should be done as quickly as possible. Samples of potash and similar substances,

which are hygroscopic, but do not attack the human skin in the same degree as caustic alkalies, are best taken by pushing the hand as deeply as possible into the barrel.

In taking samples of caustic alkalies care must be chiefly had to break up large pieces and take portions from the centre, or to scrape off the external crust and use the inner portions. This is necessary, because the surfaces of caustic alkalies generally undergo considerable changes by the absorption of moisture and carbonic acid and their consequent conversion into carbonates. It must further be taken into consideration that the alkalies do not always show the same content in all portions of the drums. This is due to the fact that the foreign salts, etc., which contaminate the product are not separated uniformly with the congealing alkali, but remain longer in solution and accumulate in the central portion of the drum which congeals last. The best average sample is, therefore, obtained by taking pieces from the bottom and sides of the drum.

## CHAPTER VIII.

## AUXILIARY RAW MATERIALS.

*Water.*—Water ( $H_2O$ ) in the manufacture of soap performs a prominent and indispensable part and may well be called a raw material. Nearly three-quarters of the earth's surface is covered by water, and it occurs chemically fixed in many minerals, most organic substances, etc. Its revolution is as follows: The water of the sea evaporates and ascends as water-vapor into the atmosphere, where it condenses to water-bubbles, which form into clouds. The wind drives the clouds over the mainland, where the water-bubbles combine to water-drops, which fall upon the earth as rain, snow, hail, etc. This so-called meteoric water collects in springs, creeks, and rivers, whence it is reconducted to the sea.

The chemical composition of water was first demonstrated, in 1781, by Cavendish, but the discovery of the exact proportions in which oxygen and hydrogen unite in generating that most important compound has, from time to time, occupied the attention of some of the most distinguished cultivators of chemical science. Nine parts of water consist of 1 part hydrogen ( $\frac{1}{9}$ ) and 8 parts oxygen ( $\frac{8}{9}$ ), or 100 parts of water contain 11.1 per cent. of hydrogen and 88.9 per cent. of oxygen.

The physical properties of water are too well known to need a lengthened description. When pure it is tasteless and inodorous, and colorless in small bulk, but has a greenish or bluish color when viewed in bulk. It is an exceedingly bad conductor of electricity of low tension. It attains its greatest density towards  $40^{\circ}$  F., freezes at  $32^{\circ}$  F., expanding thereby by  $\frac{1}{11}$  of its volume, and boils under the ordinary atmospheric pressure at or near  $212^{\circ}$  F. It evaporates at all temperatures.

Water seldom or never occurs in nature in a perfect state of



purity. Rain-water, which is the purest form of natural water, contains traces of ammoniacal salts and other impurities which it collects from the atmosphere during its descent. As soon as it reaches the earth it begins to dissolve small portions of various solid materials with which it comes in contact. The waters of wells, springs, and rivers differ very much from each other, according to the nature of the layers of rock and earth, over or through which they have passed, and from which they dissolve a great variety of substances, such as sodium sulphide and chloride, magnesium sulphate and carbonate, calcium sulphate and carbonate, carbonic acid, silica, etc.

Of the above constituents held in solution by water the carbonates and sulphates of lime and magnesia are the most injurious ones for most purposes.

Household experience has established a classification of the waters from natural sources into *soft* and *hard* waters, a division which depends chiefly upon the manner in which they act upon soap. Hard water yields a lather with soap only after the whole of the calcium and magnesium salts have been thrown down from the water in the form of an insoluble lime-soap. This, of course, causes a considerable loss in large establishments where a great deal of soap is used, since the lime-soaps formed being insoluble substances act neither mechanically nor chemically.

This peculiar property which water acquires by the presence of calcium and other salts is termed *hardness*. The simplest means to determine the hardness of a natural water is by a solution of 1 part of pure fatty acid soap in 100 parts of highly rectified alcohol, and adding 150 parts of water. By adding some of this reagent to distilled water the mixture remains clear, and on shaking yields a lather which stands four or five minutes. If the water, however, contains lime or magnesia, or both, a turbidity caused by calcium or magnesium oleate appears, which is the greater the larger the amount of these mineral substances in the water; but with very hard water caseous flakes are formed. On shaking, no permanent lather is formed until sufficient of the above-mentioned soap solution has been added to decompose the whole of the calcium and magnesium salts, and the soap solution somewhat predominates.

The *degree of hardness* is best determined by the test discovered by Clark in 1841, and improved by Liebig, Boutron, Boudet, Wilson, Faist, and Knauss.

Since the composition of soaps is not constant, the preparation of a solution of the prescribed concentration is impossible by merely weighing off a certain quantity of soap. The only way of obtaining the solution is to titrate a soap solution with calcium chloride and correct it by dilution. Soda soap having the property of gelatinizing, potash soap is preferred for the purpose, and, as an aqueous solution is subject to putrefaction and formation of mould, an alcoholic one is kept in stock. Absolute purity of the soap is not required, and as its titer is not determined by weighing, any artificial potash-soap yielding a strong lather can be used. Dissolve 10 grammes of the soap in 1 liter of dilute alcohol, filter, if necessary, and check and standardize it by a solution containing exactly 0.12 gramme of calcium oxide in 1000 cubic centimetres. This solution is obtained by dissolving exactly 0.214 gramme of pure calcite in hydrochloric acid, evaporating the solution to dryness, redissolving the residue in water, placing it in a liter flask and filling up to the liter mark on the neck. 100 cubic centimetres contain 0.012 gramme = 12 milligrammes of lime, which is considered the highest content of lime in natural water, and is designated 24 *degrees of hardness*. By dividing these 12 milligrammes into 24 degrees, each degree corresponds, of course, to 0.0005 gramme =  $\frac{1}{2}$  milligramme.

The soap solution has to be so standardized by proving that 45 cubic centimetres of it exactly correspond to 100 cubic centimetres of the lime solution; if less is required, it is diluted with alcohol; if more, some of the concentrated solution is added.

The soap solution thus checked and standardized serves for the determination of unknown quantities of lime in the water to be examined. By this method other substances which decompose soap, such as iron, magnesium, etc., are determined as lime, which is, however, admissible, since the hardness of water depends also on these constituents, and the quantity is not designated lime, but hardness.

Of this soap solution is required for 100 cubic centimetres of water containing lime—

Soap solution. Cubic centi- metres.	Lime. Grammes.	Degree of hardness.	Soap solution. Cubic centi- metres.	Lime. Grammes.	Degree of hardness.
1.4*	—	0	26.2	0.0065	13
3.4	0.0005	1	28.0	0.007	14
5.4	0.001	2	29.8	0.0075	15
7.4	0.0015	3	31.6	0.008	16
9.4	0.002	4	33.3	0.0085	17
11.3	0.0025	5	35.0	0.009	18
13.2	0.003	6	36.7	0.0095	19
15.1	0.0035	7	38.4	0.01	20
17.0	0.004	8	40.1	0.0105	21
18.9	0.0045	9	41.8	0.011	22
20.8	0.005	10	43.3	0.0115	23
22.6	0.0055	11	45.0	0.012	24
24.4	0.006	12			

\* Consumed, according to experience, by the water up to the formation of lather.

The degrees of hardness are not simply proportional to the consumption of soap solution.

Waters with an excess of lime, *i. e.*, containing more than 0.12 gramme in the liter, yield from the beginning a caseous precipitate, and long before the end of precipitation a heterogeneous membranous lather which readily gives rise to errors. Such waters must, therefore, be previously diluted with a determined quantity of distilled water, and calculated, after the consumption of the cubic centimetres, to 100 cubic centimetres of unchanged water.

The hardness of the water depends on the presence of lime, magnesia, etc.; it is, however, independent of the form in which these substances are present in solution. The direct determination of the degrees of hardness of a water gives, therefore, the *total hardness*. Lime salts kept in solution in water by the carbonic acid, which all natural waters contain, are deposited on boiling by the carbonic acid being expelled. The hardness depending on such lime is called *temporary hardness*. That quantity of lime fixed on sulphuric, hydrochloric, nitric acids, etc., which cannot be remedied by boiling, is termed *permanent hardness*.

By subtracting the permanent hardness from the total hardness, the temporary hardness is obtained.



In Germany 1 degree of hardness is = 1 part of the total lime, incl. magnesia, in 100,000 parts of water.

In England 1 degree of hardness is = 1 part in 125,000 parts of water = 1 grain in 70,000 grains of water = 1 gallon.

In France 1 degree of hardness is = 1 part in 178,571 parts of water = 1 part of calcium carbonate in 100,000 parts of water.

5 English degrees of hardness = 4 German degrees.  
100 French “ “ = 56 “ “

Or,

	German.	English.	French.
1 German degree of hardness	= 1	1.25	1.70
1 English “ “	= 0.8	1	1.43
1 French “ “	= 0.56	0.7	1

	English degrees of hardness.
<i>Soft</i> is a water of . . . . .	1.6 to 3.2
<i>Quite hard</i> is a water of . . . . .	4 to 6.4
<i>Hard</i> is a water of . . . . .	6.4 to 7.2
<i>Very hard</i> is a water of . . . . .	9.6

By this decomposition 31 parts of sodium oxide or 47 parts of potassium oxide are replaced by 28 parts of calcium oxide or 20 parts of magnesium oxide, hence 1 degree of hardness annihilates about  $0.01 = 1$  centigramme of ordinary soap, or 1 liter of water of 20 degrees hardness decomposes 2 grammes, and 1 cubic metre of the same water, 2 kilogrammes (4.4 pounds) of soap.

Now suppose that, generally speaking, the loss of soap by the hardness of water amounts to  $\frac{1}{3}$  to  $\frac{1}{4}$ , this, with the extensive consumption of soap and its value, represents enormous sums. The city of London consumes annually over 12,000 tons of soap, which, at £50 sterling per ton, makes a total of £600,000 sterling; hence, if all the water were hard, £150,000 sterling would be lost, and if two-thirds of the water were hard £100,000 sterling.

It is, however, not only the direct loss of soap which comes hereby into question; but in washing the magnesia and lime soaps formed close up the pores of the skin and form a deposit on the fibres of the washed clothes, especially wool, so that in drying they lose their softness and acquire a bad odor.

A simple remedy for such hard waters is a solution of silicate

of soda of 20° B., 5 per cent. of which added to the water will cause the lime, etc. to precipitate and leave the water sufficiently pure for use. In the preparation of alkaline lyes this is effected spontaneously as the free carbonic acid in the lime is precipitated on causticizing with lime.

For technical purposes turbid and impure water can be purified by standing and subsequent filtering through sand. Substances dissolved in the water are, however, not removed by filtration. Perfectly pure water can only be obtained by distillation.

The following simple methods for testing the purity of water are published by the "Centralblatt für Textil Industrie," and as they may be of interest to soap-boilers, we give them here:—

1. For testing whether water is *hard* or *soft*, some white soap is dissolved in alcohol, and a few drops of the solution are added to the glass of water to be tested. If this becomes milky, the water is hard; if it is not changed, or is only troubled in a slight degree, it is soft.

2. For discovering the presence of *copper*, filings of soft iron are introduced into the water and left there for a few minutes, after which time a couple of drops of spirit of sal-ammoniac are added. A blue tinge denotes the presence of copper.

3. For discovering *carbonic acid*, some water is mixed with an equivalent quantity of lime-water. If carbonic acid be present, the mixture immediately becomes milky. This disturbance is removed by the addition of muriatic acid.

4. *Combinations of sulphur* are discovered by a little quicksilver being introduced into the water, which has been placed in a bottle. The bottle is then closed and allowed to stand for several hours. If the quicksilver assumes a darker surface, and if, on being shaken, it separates into a silver-gray powder, it is a sign that the water contains combinations of sulphur.

5. *Dissolved pure lime* is detected if the introduction of one or two crystals of oxalic acid into the water produces a milky deposit.

6. *Sulphate of lime* is recognized if in the solution by chlorine of barium a white deposit takes place, which is not again dissolved in nitric acid.

7. *Alkalies and alkaline earths* are discovered in the following manner: Litmus paper is dyed a weak red by diluted vinegar, and is plunged into the water to be tested. If the previous blue shade be restored, the water is of an alkaline nature.

8. The presence of *iron* is recognized by several drops of a decoction of gall-nuts, which are added to a glass of the water. If iron be present, the water assumes an inky appearance, varying between a gray and a black color. Water containing iron is likewise colored blue by a drop of a solution of ferrocyanide of potassium.

9. *Magnesia* is detected by the water being heated to the boiling point, and by a small quantity of carbonate of ammonia and phosphate of soda being added (enough to go on the point of a knife). If magnesia be present, it is precipitated at the bottom of the vessel.

10. *Free acids* are discovered by plunging a small piece of blue litmus paper into the water. A red tinge denotes the presence of free acids. If in such acid water a white precipitate is caused by lime water, then carbonic acid has been present.

*Lime.*—Burnt lime or quick lime ( $\text{CaO}$ ) is the monoxide of the metal calcium. Neither the metal itself nor its oxide occurs as such in nature, though compounds of the metal with acids are very abundant and are widely distributed. Among them may be enumerated the carbonate, which exists as chalk, limestone, marble, calc-spar, etc., the sulphate as gypsum and selenite, the fluoride as fluor-spar, and the phosphate as apatite and bone-earth.

Water containing carbonic acid coming in the interior of the earth in contact with lime salts, becomes hard water by the absorption of calcium carbonate and sulphate. From the water the lime combinations reach the plants, and from them and the water the animal organism. The ash of plants contains calcium carbonate, phosphate, and sulphate; the shells of eggs, oysters, etc. consist almost entirely of calcium carbonate; and bones contain about half their weight of calcium phosphate and carbonate.

By heating calcium carbonate ( $\text{CaCO}_3$ ) to from  $1140^\circ$  to  $1482^\circ$  F. the carbonic acid is expelled, while calcium monoxide remains behind. Upon this is based the process of lime-burning. With



too high a temperature the small quantity of silica and alumina contained in the limestone becomes sintered over the surface, which prevents the escape of the carbonic acid; such lime is called *overburnt*. Notwithstanding the considerable loss of weight (over 40 per cent.) the calcium carbonate suffers by burning, its bulk does not decrease to any extent, the burnt lime remaining behind as a porous mass. The freer the lime-stone from foreign admixtures the better the lime produced from it. When the lime-stone consists chiefly of pure carbonate of lime, the resulting product is, what is termed, "*fat lime*." On the other hand, if the limestone contains magnesium carbonate (dolomite  $\text{CaCO}_3\text{MgCO}_3$ ) the resulting lime forms a short, thin pulp with water and is termed "*poor*."

Pure lime is white and often of considerable hardness; the ordinary commercial article, however, has generally a grayish-yellow color and contains some alumina, ferric oxide, etc. It rapidly combines with water under the development of considerable heat. If very little water is added, it breaks up and decomposes to a soft, white powder, with a burning and caustic taste, which in bulk exceeds three times that of the lime used. This conversion is known as "slaking" and the product as "slaked lime" or calcium hydroxide ( $\text{CaH}_2\text{O}$ ). By pouring water on slaked lime a more or less thin paste is obtained, which is called milk of lime. It is always in this form that lime is used to prepare caustic lyes of potash and soda.

Burnt lime exposed for some time to the air absorbs moisture and carbonic acid; being thereby converted into carbonate of lime. In this state it has lost all its causticity, and does not possess the property of depriving the carbonate of potash and soda of their carbonic acid.

Lime is less soluble in hot water than in cold, 1 part of lime requiring 763 parts of cold water and 1270 parts of hot water, or 100 parts of water dissolve at an ordinary temperature 0.70 part of lime and only 0.12 part when heated.

It is recommended to keep lime in tight barrels covered with ashes to prevent the access of atmospheric air. A very good method for keeping lime for use is to slake it into a stiff paste and put it into water-tight vats or barrels, where it will keep for

a long time; the upper surface alone having absorbed carbonic acid can be removed before using the lime for causticizing the lye.

The value of lime for the manufacture of soap can be determined in the following manner: Slake 10 grammes of the lime with water in a dish, pour a 10 per cent. sugar solution over it, rinse the whole into a one-half liter flask, fill up the flask to the one-half liter mark with a 10 per cent. sugar solution, and allow the whole to stand 24 hours with frequent shaking. Then pass the whole through a filter, take 50 cubic centimetres of the filtrate, color it blue with litmus tincture and titrate with normal sulphuric acid until the liquid acquires a red coloration. By multiplying the cubic centimetres of acid used with  $0.028 \times 10$ , the per cents. of caustic lime contained in the sample are obtained. Good limestone should not contain less than 82 per cent. of calcium oxide, and, if possible, over 86 per cent.

*Common salt.*—Common salt, or, as it is called by the chemist, sodium chloride ( $\text{NaCl}$ ), is of great importance for all industries. It is the source of nearly all the soda used for technical purposes, of all the chlorine, hydrochloric acid, chloride of lime, and all products containing chlorine.

In nature common salt is found in the solid, as rock salt, as well as dissolved in the water of some springs and the sea, in enormously large quantities. It is obtained either by evaporation of its solutions (sea water and water of salt springs), or, where it occurs in extensive layers as rock salt, in large blocks, by mining. The following are a few localities where extensive deposits of rock salt are found: In the Prussian provinces of Brandenburg, Saxony (Stassfurt and Erfurt), Posen and Schleswig-Holstein; in Bavaria (Berchtesgaden); in Upper Austria (Salzburg, Hall, Hallein, Ischl); in Galicia (Wieliczka) and Transylvania; in Spain (Catalonia); in England; and the United States.

Rock salt frequently occurs colored red, or blue, or brown, and is decolorized by dissolving and clarifying.

The following analyses show the composition of rock salt:—

	White, from Wieliczka and Stassfurt.	Yellow, from Berchtes- gaden.	Reddish, from Hall in Tyrol.	Detonating salt,* from Hallstadt.
	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>
Sodium chloride . . .	100.00	99.85	99.43	98.14
Potassium chloride . . .	—	—	—	trace
Calcium chloride . . .	—	trace	0.25	—
Magnesium chloride . . .	trace	0.15	0.12	—
Calcium sulphate . . .	—	—	0.20	1.86
	100.00	100.00	100.00	100.00

\* The so-called detonating salt found in crystalline granular masses has the property when being dissolved in water of giving rise to slight detonations, accompanied by an evolution of hydrocarbon gas from microscopically small cells, the walls of which, becoming thin when the salt is dissolved in water, give way, and cause the report.

Perfectly pure common salt is not hygroscopic, but is made so by admixtures of magnesium and calcium chloride contained in the salt of commerce. Usually salt contains from 2.5 to 5.5 per cent. of water, not as a constituent but as an intermixture, hence the phenomenon called decrepitation, due to the breaking up of the crystals by the action of the steam when salt is heated. Ignited to a strong red heat common salt fuses, forming an oily liquid, and at a white heat is volatilized without decomposition.

Common salt dissolves with almost equal ease in cold and hot water, a saturated solution containing nearly 27 per cent.

According to Poggiale, 100 parts of water dissolve of sodium chloride—

32.73 parts at . . . . 50° F.	36.64 parts at . . . . 104° F.
33.49 “ . . . . 14	37.25 “ . . . . 140
34.22 “ . . . . 23	37.88 “ . . . . 158
35.52 “ . . . . 32	38.22 “ . . . . 176
35.63 “ . . . . 41	38.87 “ . . . . 194
35.74 “ . . . . 48	39.61 “ . . . . 212
35.87 “ . . . . 57	40.35 “ . . . . 229.5
36.13 “ . . . . 77	

The following table shows the specific gravity of various solutions of common salt:—



*Sodium chloride solutions.*

Specific gravity.	Per cents.	Specific gravity.	Per cents.	Specific gravity.	Per cents.	Specific gravity.	Per cents.
1.00725	1	1.05851	8	1.11146	15	1.16755	22
1.01450	2	1.06593	9	1.11938	16	1.17580	23
1.02174	3	1.07335	10	1.12730	17	1.18404	24
1.028999	4	1.08097	11	1.13523	18	1.19228	25
1.03624	5	1.08859	12	1.14315	19	1.20098	26
1.04366	6	1.09622	13	1.15107	20		
1.05108	7	1.10384	14	1.25931	21		

$$1.20344 = 26.395 \text{ per cent.}$$

In boiling soap common salt is so important a material for refining, that the soap-maker should pay proper attention to procure it reasonably pure, otherwise he may fail to obtain a good color, or he may lose by the decomposed soap and alkali carried off with the waste lye. Salt, if necessary, may be refined by dissolving it in a suitable quantity of warm water and adding solution of silicate of soda in the proportion of about five per cent., when, after stirring for some time and being left to rest, it will carry down with the precipitate almost all the lime and other salts, and the soap-maker may use the upper clear portion with confidence. By means of crystallized sodium carbonate, a solution of common salt can also be somewhat purified, as the contaminating salts are thereby changed into insoluble carbonates which precipitate and can be readily separated.

*Soluble glass or water glass. Silicate of soda, silicate of potash.*—Soluble glass or water glass is a combination of silicic acid and alkalis. Prof. Fuchs, of Munich, demonstrated in 1818 that glass could be dissolved in water and called this liquid glass “water glass.”

There are in Germany at the present time eight manufactories of soluble glass, with a market for their product, not only in Germany, Austria, and Italy, but also in Russia, Sweden, Denmark, and a large portion of the United States.

Soluble glass is obtained either from infusorial earth and lye by the so-called wet process, or from fine white siliceous sand and calcined soda or potash by the fusing process.

The first process chiefly consists in bringing in a tapering wooden vessel, lye to the boiling point, by means of steam, and gradually adding with uninterrupted stirring infusorial earth, which dissolves after continued boiling, and yields soluble glass of from  $28^{\circ}$  to  $30^{\circ}$  B.; 240 to 250 pounds of infusorial earth suffice for 100 pounds of lye.

In most factories the soluble glass is at present prepared by fusing together from 44 to 45 pounds of the finest white siliceous sand, 3 parts of pulverized charcoal, and 22 to 23 parts of calcined soda or potash.

The process is briefly as follows: The substances are first intimately mixed and then brought into a furnace, where they fuse to a liquid mass in from eight to ten hours. The mass is then drawn off and allowed to cool to lumps of glass weighing frequently from 30 to 50 pounds. This glass is then comminuted, ground to a powder by suitable rolls and sifted, and then forms the water-glass powder; it is shipped in this state in order to save freight. It readily dissolves in twice its quantity of water heated by steam; it is, however, necessary to use a double boiler, or a conical, funnel-shaped wooden vat, to prevent the powder from settling on the bottom: 100 pounds of the powder yield about 300 pounds of soluble glass of  $30^{\circ}$  to  $40^{\circ}$  B.

Potassium silicate is employed for filling soft soaps, and sodium silicate for bar soaps. Soluble glass is as clear as water and has a specific gravity of 1.3.

## CHAPTER IX.

## LYES.

*Preparation of lyes.*—The preparation of lyes, *i. e.*, solutions of caustic soda or caustic potash, is the first operation required for the manufacture of soap. Lyes are of twofold importance: they form, on the one hand, an actual constituent of the finished soap; and, on the other, constitute the substance on whose chemical action depends that conversion of the fats which must precede the formation of soap. For the preparation of lye from caustic alkalies no special arrangements are required, though the breaking out of the caustic soda from the drums being very troublesome work, many contrivances are resorted to, to dissolve the soda in the drum itself. One plan much in use is to place the drum, after taking out the head and bottom, upon an elevated grating in a kettle, pouring water over it and heating. In a few hours the caustic soda is dissolved, the empty drum taken out, and the solution brought to the desired degree. After drawing off the clear lye into tanks, water is added to the slimy mass remaining in the kettle. This yields a weaker lye, which is used in boiling grain-soaps. It is best to dissolve at one time as much caustic soda as will furnish sufficient lye for working the quantity of fat to be used in a week or a month. An allowance of 25 pounds of caustic soda for 100 pounds of fat is ample, so that for a factory where 20,000 pounds of fat are worked into hard soap in a month, 5000 pounds of caustic soda will have to be dissolved. A very large kettle being required for the solution of such a quantity, it is preferable to dissolve one-fourth or one-fifth of it at a time. Sufficient water should only be used for the solution to show at least 40° B. For each 100 pounds of caustic soda add 200 pounds of water, and admit steam or start the fire under the kettle. As soon as the water becomes hot, the soda dissolves

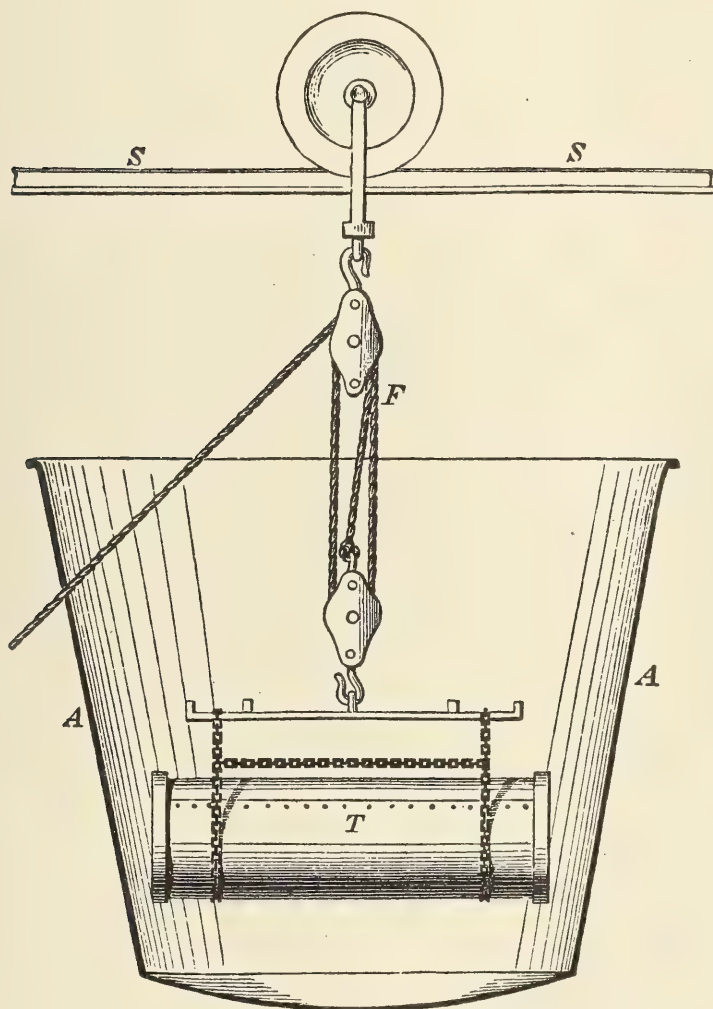


without the necessity of stirring. The empty drums are then taken out and rinsed off with weak lye.

In four or five days, when all the foreign salts have crystallized out, the clear lye is drawn off into tanks, and by the addition of water brought to the degree required.

The residue in the kettle, which is often thick and turbid and contains the foreign salts, is diluted with water and heated by steam or fire and brought to  $20^{\circ}$  B. After settling over night,

Fig. 26.



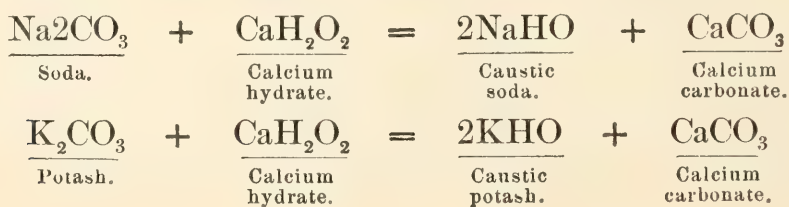
the clear lye is drawn off and is used for the saponification of cocoanut or palm-kernel oils. The residue is diluted with water to  $1^{\circ}$  B., the weak lye thus obtained being used for dissolving the next lot of caustic soda.

A suitable arrangement for dissolving the caustic soda in the drum itself is shown in Fig. 26. An iron rail, *S*, is secured in walls of the factory so as to run about  $6\frac{1}{2}$  to  $7\frac{1}{2}$  feet above the edge, and directly over the centre of the kettle *A*. Upon this rail runs a large cast-iron pulley (about 12 inches in diameter), to which is fastened the tackle *F*. The lower end of the tackle is provided with a strong iron hook.

The drum to be emptied is first pounded on all sides with a heavy iron hammer, which causes the soda to crack. Holes the size of a hand are then cut with a hatchet in the head and bottom of the drum near the edges. Two chains are next slung around the drum and suspended to a horizontal iron rod, as shown in the illustration. The drum is then hoisted to above the edge of the kettle by means of the tackle, and, after pushing forward the pulley upon the iron rail to directly over the centre of the kettle, the drum is lowered to about one-half the height of the kettle and the tackle secured. Water is then admitted to the kettle, and the drum from time to time raised to permit the dissolved soda to run out. The entire work, including the dissolving of the soda, is effected at the utmost in two hours, about ten minutes of that time being taken up by preparing and placing the drum in position.

The preparation of lyes from the carbonates of the alkalies requires more care and attention. The carbonates found in commerce in the form of wood-ash, potash, and soda do not decompose the fats (with the exception of rancid fats, such as cocoanut-oil and palm-oil) even at a boiling heat, so that, to render them effective, they have to be deprived of their carbonic acid with the assistance of lime.

In causticizing the carbonates of the alkalies by means of burnt lime, a combination of great chemical activity is formed by a simple exchange; the carbonic acid of the alkali enters the lime, while the alkali takes up the hydroxide of the lime.



This chemical process takes place, however, only in treating dilute solutions of the carbonates of the alkalies. A concentrated lye of caustic soda or caustic lye again withdraws the carbonic acid from the calcium carbonate, the result being a reaction which is just the reverse of what the soap-boiler has in view. This explains the important fact taught by experience, that potassium carbonate treated with 4 parts of water is not rendered caustic at all, and only incompletely so with 5 to 8 parts, but is completely causticized when dissolved in more than 10 parts of water.

The best and quickest method of effecting causticity is to gradually add slaked lime to a boiling hot solution of soda or potash until a sample clarified by settling no longer effervesces when poured into dilute acid, which proves that all the carbonic acid has passed into the lime and that the lye is perfectly caustic.

Theoretically, *i. e.*, with perfectly pure materials, 100 parts of potash require 40.58 parts of lime to be rendered completely caustic, and 100 parts of soda 52.83 parts of lime, because—

138	:	56	=	100 : X ; X = 40.58 lime.
Atomic weight of potash.		Of lime.		
106	:	56	=	100 : X ; X = 52.83 lime.
Atomic weight of soda.		Of lime.		

It is, however, found that even with the use of chemically pure materials these quantities do not suffice, since pure calcium carbonate is not formed, it entering into a loose combination with the undecomposed calcium hydrate ( $\text{CaCO}_3, \text{CaH}_2\text{O}_2 =$  basic calcium carbonate or calcium hydrocarbonate), so that only three-quarters of the quantity of lime used is effective. Hence, a larger quantity than the theoretical one has to be used.

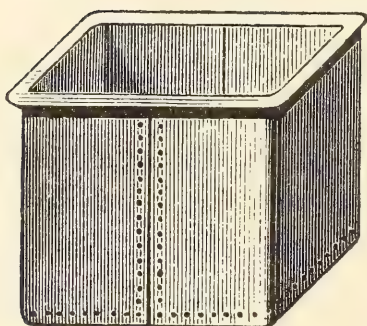
The carbonates of the alkalies are also rendered caustic by treatment in the cold, the process requiring, however, a greater quantity of lime, more dilute solutions, and more time.

For the preparation of lyes from the carbonates of the alkalies, the potash or soda solutions are generally treated with milk of lime in large square tanks of sandstone or cast or wrought



iron. These tanks, Fig. 27, are generally about  $6\frac{1}{2}$  feet high and wide, and are not provided with a perforated bottom. They are frequently inserted to about half their height in the ground, or project into the cellar where they rest upon columns. The calcined soda used has the tendency of passing into crystallized soda on coming in contact with water; it settles on the bottom of the tank, whereby the crystals formed envelop the remainder of the undissolved, anhydrous salt, and form lumps, which can only be dissolved with difficulty by long and constant stirring. To avoid

Fig. 27.



this, the soda is best placed in a perforated iron box, which is suspended in the tank so as to be even with the level of the water in the manner shown in Fig. 26. By the water penetrating the perforations, concentrated solution of soda is constantly formed, which, being specifically heavier than water, sinks to the bottom, making room for non-saturated quantities of water until all the soda is dissolved. A similar treatment of

potash is recommended, since, though it is readily soluble, grosser contaminations, such as stones, etc., remain in the box.

When solution is complete the necessary quantity of milk of lime is added, and, after frequent stirring, the whole is allowed to rest to permit the calcium carbonate, etc. to settle. The first lye is then drawn off and lixiviation continued with fresh water.

Messrs. Charles Tennant & Co., the extensive alkali manufacturers of St. Rollox, Glasgow, give the following directions for the preparation of lye: A layer of fresh burnt lime—say 5 measures of 112 pounds each—is to be laid equally over the bottom of the vat, and a few gallons of water to be thrown upon the lime until it begins to slake or fall. This layer is then to be covered immediately with six hundredweight of soda ash, the next layer with four measures of lime slaked as before, the fourth layer with the same quantity of soda ash, the fifth layer with lime as before, and the last layer with the same quantity of alkali.

After standing two hours, the vat is to be stanchd by filling it with water or weak lye of a former vat; this is to be done gradually. After standing about fifteen or sixteen hours, the plug is to be gently loosened, so as to allow the lye to run off or trickle clear and caustic after infiltration through the beds of lime. This is called the *first runnings*. As soon as the lye ceases to run the plug is to be tightened and the vat again filled with water, and after standing a sufficient time, to be run down as before. This is the *second runnings*, and, worked together with the first runnings in the soap-kettle, is an excellent lye and works freer and better than if used separately. After the vat is run dry the contents are turned over into another vat, covered with water, and again run down. This lye is very weak and is seldom worked in the soap-kettle, being used instead of water to stanch or fill up the strong or first-set vats. As soda ash is not all equally soluble, it is sometimes necessary to turn the contents of the vat over a second time in order to obtain all the free alkali; but experience and care are the only sure guides. The receivers of the lye are generally much smaller vats, but it is preferable to have them of the same size, it being at all times desirable to have a sufficient supply of strong caustic lye.

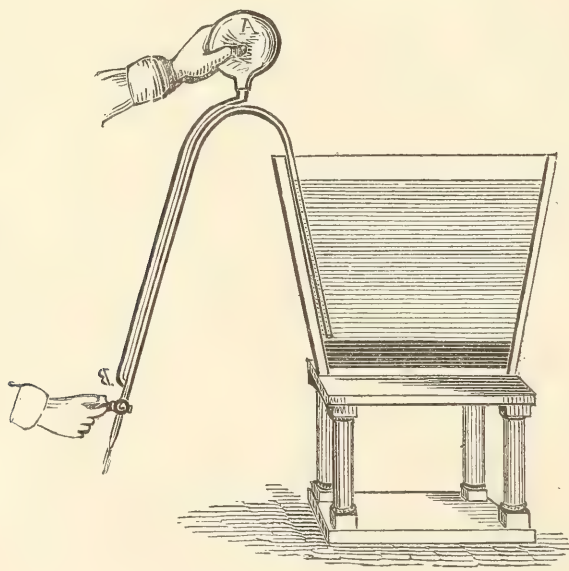
In factories working with steam, the mixture of milk of lime and soda solution is heated to the boiling point by the introduction of steam through an iron pipe running to the bottom of the tank. By these means the solution is more quickly and more completely causticized, and the calcium carbonate formed settles with greater ease and forms a denser deposit than in preparing lyes in the cold way. Besides, the more granular precipitate is lixiviated with greater ease, so that the loss of alkali is also smaller.

A tank of the dimensions named ( $6\frac{1}{2}$  feet high and wide) holds about 282 cubic feet or about 1780 gallons of water, and since soda dissolved in 8 parts of water completely yields its carbonic acid, 2200 pounds of pure anhydrous soda or 2420 pounds of 90 per cent. soda can be employed at one time; of potash, however, only two-thirds that quantity, since it is only completely causticized when diluted with 12 parts of water. The tank is filled half full with water, and after suspending the per-

forated iron box containing the soda or potash in the tank, the liquid is heated by the introduction of steam. Solution is effected so quickly that the total quantity of material—fresh portions of which are from time to time introduced into the box—is dissolved by the time the liquid has reached the boiling point. When this is the case, milk of lime is admitted from a reservoir placed at a higher level, or, what is still better, the required quantity of unslaked lime is gradually brought into the perforated box. In the hot soda or potash solution the lime slakes very rapidly and runs as milk of lime through the perforations of the box, while the portions remaining unslaked are left behind in the box. A sample is then taken and tested for carbonic acid in the manner described on page 223. If no effervescence takes place, the addition of lime is interrupted, the steam shut off, and after filling the tank with water and thoroughly stirring the whole several times, the tank is covered with a lid.

The next day, the calcium carbonate having settled, the clear lye is dipped out with a ladle or drawn off by means of a siphon.

Fig. 28.



The siphon (Fig. 28) should be of half-inch lead pipe, and consists of a bent tube, one leg of which is longer than the other, and is provided on the lower end with the cock *B*. Above the



cock a narrower tube, capped with a hollow rubber ball *A*, enters the leg. By immersing the short leg of the siphon in the clear lye to within an inch or less of the deposit of calcium carbonate and squeezing the rubber ball, the cock being open, the air in the siphon is rarefied and the denser outer air forces the liquid up to the highest point of the bend, whence it drops by the force of gravitation and flows out in a continuous stream as long as the mouth of the short leg is covered by it.

After drawing off the clear lye, water is poured over the deposit of lime, with thorough stirring, until the tank is about half full. After allowing the mixture to rest, the clear lye is drawn off into a second tank and used for dissolving and causticizing fresh quantities of soda or potash. Water is then again poured over the residue in the first tank, the whole stirred, and clarified by resting. This weak lye serves for washing the residue in the second tank. The residue in the first tank, when entirely freed from lye, is removed and fresh lye set in the tank with the wash-water from the third tank. The fresh lye is always prepared boiling hot; cold water, however, being used for further lixiviation.

By the hot method lye of medium concentration only is obtained, which, if required of greater strength, is evaporated in kettles. The lyes prepared by the hot process have the advantage of being entirely caustic, *i. e.*, free from carbonic acid; moreover, no further loss of alkali takes place.

A material disadvantage of the cold way of preparing lye is, that the action of the caustic lime cannot previously be accurately determined, since the calcium carbonate formed withdraws sometimes more and sometimes less caustic lime, the calcium hydrocarbonate, mentioned on page 223, not being a constant chemical combination. This is the reason why such lye is at one time nearly caustic and at another time too caustic, *i. e.*, contains too much lime, or, as it is technically called, is *too high* in lime, or contains much alkaline carbonate, *i. e.*, is *too low* in lime. No disadvantage is connected with the first two cases, but when the lye still contains carbonic acid, nothing is left but to pour it again over lime or to use it for cocoanut or palm-kernel oils, which saponify more readily on account of the free fatty acids

they contain, or to employ it for grained soft soap to facilitate crystallization. With nearly caustic lyes, or those too high in lime, the quantity of fat to be saponified can be readily calculated, which, however, is not the case without a previous test (determination of carbonic acid) with lyes containing alkaline carbonate. Hence, if a content of alkaline carbonate is required for the success of the soap, it is better to add it to good lye. It is further known that the carbonates of the alkalies do not act upon the fats, and in salting out the soap pass unchanged into the salt-lye. In paste-soaps the alkaline carbonate remains suspended in the form of an emulsion and is of no value to the consumer, but rather an injury, since in washing it dissolves first, and, if present in excess, attacks the hands, fibre of the clothes, etc.

For paste-soaps, lye containing much potassium and sodium chloride and sulphate cannot be used; such lye must first be concentrated to a certain degree in order to separate the greater portion of the mentioned salts.

In this country the sodium aluminate obtained in the manufacture of soda from cryolite (p. 179), and known as "*natrona refined saponifier*," is used for saponification together with caustic soda.

*Preparation of lye from wood-ashes.*—The ash, freed by sifting from pieces of charcoal and semi-carbonized wood, is mixed as thoroughly as possible with the required quantity of lime slaked to powder. The mixture is placed in a wooden, or still better in an iron vat in which is inserted a sieve bottom covered with straw. Sufficient water is then added so as to form a thick paste, and the whole allowed to rest twenty-four hours. It must be observed that the layer is everywhere of equal height, and no gutters are formed through which the water might flow off without having previously absorbed the caustic potash. Now pour water into the yet empty part of the vat and permit the lye to draw off. The space between the sieve and actual bottom must have an opening immediately under the former so that the air can escape. The lye which collects between the two bottoms is drawn off by means of a stop-cock and carried to a second vat, which is prepared in the same manner as the first. It may also be passed into a third vat, but in every case the quantity of water must be so

proportioned that only lyes of 7 per cent. caustic potash are produced. Lyes of this strength may be used for the saponification of the fats. They contain, however, as a rule, such large amounts of potassium sulphate and chloride that muddy soaps would be obtained. It is therefore necessary to condense them to  $22^{\circ}$  to  $25^{\circ}$  B., when, after cooling, the greater portion of these foreign salts crystallizes out. For use the lyes are again diluted with water till they reach the desired strength.

*Testing the lyes.*—In most soap factories the lyes are tested by means of an areometer, which is a very convenient method, but gives rise to great errors. The areometer simply indicates the specific gravity of the lyes, which depends on all the substances dissolved in them, while the soap-boiler only desires to know the content of potash or soda. If the soap-boiler had only to deal with solutions of pure soda or potash, testing with an areometer would suffice, but as the lyes contain a quantity of combinations of chlorine, sulphates, etc., two lyes showing the same degree by the areometer may contain varying quantities of caustic alkali or of alkaline carbonates.

The principal areometers used are Baumé's and Twaddle's, the latter being chiefly employed in England.

The following tables give a comparison of Baumé's degrees with those of Twaddle, and, at the same time, indicate the different per cents. of various alkalies for a more simple calculation of the quantities required for the preparation of lyes:—



*Table for the comparison of Baumé's degrees with those of Twaddle, with specific gravities and statement of the percentage in soda\* and potash lyes at 59° F.*

Degrees according to Baumé.	Degrees according to Twaddle.	Specific gravity.	Per cent. by weight, sodium carbonate.		Per cent. by weight, potassium carbonate, $K_2CO_3$ .
			Calcined, $Na_2CO_3$ .	Crystallized, $Na_2CO_3 + 10H_2O$ .	
0.0	0.0	1.0000	—	—	—
0.5	0.7	1.0035	0.33	0.90	0.35
1.0	1.4	1.0070	0.67	1.80	0.70
1.5	2.1	1.0105	1.00	2.75	1.10
2.0	2.8	1.0141	1.33	3.50	1.50
2.5	3.6	1.0177	1.71	4.62	1.90
3.0	4.4	1.0213	2.09	5.64	2.30
3.5	5.1	1.0249	2.43	6.04	2.70
4.0	5.8	1.0286	2.76	7.44	3.10
4.5	6.6	1.0323	3.08	8.84	3.55
5.0	7.4	1.0360	3.43	9.25	4.00
5.5	8.2	1.0397	3.86	10.41	4.45
6.0	9.0	1.0435	4.29	11.57	4.90
6.5	9.7	1.0473	4.66	12.45	5.30
7.0	10.4	1.0511	4.94	13.32	5.70
7.5	11.2	1.0549	5.38	14.36	6.10
8.0	12.0	1.0588	5.71	15.40	6.50
8.5	12.7	1.0627	6.04	16.29	6.90
9.0	13.4	1.0667	6.37	17.18	7.30
9.5	14.2	1.0706	6.74	18.19	7.75
10.0	15.0	1.0746	7.12	19.20	8.10
10.5	15.8	1.0787	7.50	20.22	8.50
11.0	16.6	1.0827	7.88	21.25	9.00
11.5	17.3	1.0867	8.25	22.25	9.40
12.0	18.2	1.0909	8.62	23.25	9.80
12.5	19.1	1.0951	9.03	24.34	10.15
13.0	20.0	1.0992	9.43	25.43	10.70
13.5	20.8	1.1035	9.81	26.46	11.15
14.0	21.6	1.1077	10.19	27.48	11.60
14.5	22.4	1.1120	10.57	28.50	12.00
15.0	23.2	1.1163	10.95	29.53	12.40
15.5	24.1	1.1206	11.38	30.64	12.75
16.0	25.0	1.1250	11.81	31.85	13.30
16.5	25.9	1.1294	12.22	32.95	13.75
17.0	26.8	1.1339	12.61	34.00	14.20
17.5	27.6	1.1383	13.25	35.61	14.60
18.0	28.4	1.1423	13.79	37.21	15.00
18.5	29.4	1.1474	14.21	38.86	15.50
19.0	30.4	1.1520	14.64	39.51	16.00
19.5	31.4	1.1566	15.02	40.65	16.50
20.0	32.4	1.1613	15.49	41.79	17.00
20.5	33.6	1.1660	15.83	42.89	17.50
21.0	34.2	1.1707	16.27	43.89	18.00
21.5	35.1	1.1755	16.68	44.93	18.40

\* Since from 18° B. upwards the soda solutions commence to crystallize, the statements up to 34° B. refer to 86° F.

*Table for the comparison of Baumé's degrees with those of Twaddle.—Continued.*

Degrees according to Baume.	Degrees according to Twaddle.	Specific gravity.	Per cent. by weight, sodium carbonate.		Per cent. by weight, potassium carbonate, $K_2CO_3$ .
			Calcined, $Na_2CO_3$ .	Crystallized, $Na_2CO_3 + 10H_2O$ .	
22.0	36.0	1.1803	17.04	45.97	18.80
22.5	37.0	1.1852	17.47	47.04	19.25
23.0	38.0	1.1901	17.90	48.31	19.70
23.5	39.0	1.1950	18.33	49.46	20.20
24.0	40.0	1.2000	18.76	50.62	20.70
24.5	41.0	1.2050	19.18	51.26	21.15
25.0	42.0	1.2101	19.61	52.91	21.60
25.5	43.0	1.2152	20.04	54.10	22.05
26.0	44.0	1.2202	20.47	55.29	22.50
26.5	45.1	1.2255	20.94	56.50	23.00
27.0	46.2	1.2308	21.42	57.80	23.50
27.5	47.1	1.2361	22.18	58.97	24.00
28.0	48.2	1.2414	22.39	60.15	24.50
28.5	49.3	1.2468	22.77	61.44	25.00
29.0	50.4	1.2522	23.25	62.73	25.50
29.5	51.5	1.2576	23.72	63.89	26.15
30.0	52.6	1.2632	24.18	65.24	26.60
30.5	53.7	1.2687	24.65	66.50	27.05
31.0	54.8	1.2743	25.11	67.76	27.50
31.5	55.9	1.2800	25.55	69.07	28.00
32.0	57.0	1.2857	26.04	70.28	28.50
32.5	58.2	1.2915	26.55	71.65	29.05
33.0	59.4	1.2973	27.06	73.02	29.60
33.5	60.5	1.3032	27.52	74.75	30.15
34.0	61.6	1.3091	27.97	75.48	30.70
34.5	62.8	1.3151	—	—	31.15
35.0	64.0	1.3211	—	—	31.60
35.5	65.2	1.3272	—	—	32.15
36.0	66.4	1.3333	—	—	32.70
36.5	67.7	1.3395	—	—	33.25
37.0	69.0	1.3458	—	—	33.80
37.5	70.2	1.3521	—	—	34.30
38.0	71.4	1.3585	—	—	34.80
38.5	72.7	1.3649	—	—	35.35
39.0	74.0	1.3714	—	—	35.90
39.5	75.3	1.3780	—	—	36.45
40.0	76.6	1.3846	—	—	37.00

*Table giving the content of solutions of soda\* and potash according to degrees Baumé in kilogrammes at 59° F.*

Degrees according to Baumé.	100 liters contain kilogr. soda, $\text{Na}_2\text{CO}_3$ .	100 liters contain kilogr. potash, $\text{K}_2\text{CO}_3$ .	Degrees according to Baumé.	100 liters contain kilogr. soda, $\text{Na}_2\text{CO}_3$ .	100 liters contain kilogr. potash, $\text{K}_2\text{CO}_3$ .
1	0.68	0.7	26	24.97	27.5
2	1.35	1.5	27	26.37	28.9
3	2.14	2.3	28	27.66	30.4
4	2.84	3.2	29	29.11	31.9
5	3.55	4.1	30	30.54	33.6
6	4.48	5.1	31	31.99	35.0
7	5.20	6.0	32	33.46	36.6
8	6.05	6.9	33	35.10	38.4
9	6.80	7.8	34	36.59	40.2
10	7.65	8.7	35	—	41.7
11	8.53	9.7	36	—	43.6
12	9.40	10.7	37	—	45.5
13	10.30	11.8	38	—	47.2
14	11.29	12.9	39	—	49.2
15	12.22	13.8	40	—	51.2
16	13.29	15.0	41	—	53.4
17	14.30	16.1	42	—	55.4
18	15.75	17.1	43	—	57.7
19	16.87	18.4	44	—	60.0
20	18.00	19.8	45	—	62.2
21	19.05	21.1	46	—	64.6
22	20.01	22.2	47	—	67.0
23	21.40	23.4	48	—	69.0
24	22.51	24.8	49	—	72.2
25	23.73	26.1	50	—	75.0

\* Since from 18° B. upwards the soda solutions commence to crystallize the statements up to 34° B. refer to 86° F.



*Table showing the yield of milk of lime, in degrees Baumé, from 1 kilogramme of caustic lime, according to Edward Mategcek.*

1 kilogr. of caustic lime yields milk of lime according to Baumé.		Weight of the milk of lime.	1 kilogr. of caustic lime yields milk of lime according to Baumé.		Weight of the milk of lime.
Degrees.	Liters.		Degrees.	Liters.	
10	7.50	9.44	38	3.39	5.07
11	7.10	9.01	39	3.37	5.05
12	6.70	8.60	40	3.35	5.03
13	6.30	8.20	41	3.34	5.01
14	5.88	7.80	42	3.32	5.00
15	5.50	7.43	43	3.31	4.98
16	5.25	7.16	44	3.30	4.96
17	5.01	6.92	45	3.29	4.95
18	4.80	6.70	46	3.28	4.93
19	4.68	6.51	47	3.27	4.92
20	4.42	6.38	48	3.26	4.90
21	4.24	6.18	49	3.25	4.89
22	4.16	6.05	50	3.24	4.88
23	4.05	5.92	51	3.23	4.87
24	3.95	5.81	52	3.22	4.86
25	3.87	5.72	53	3.215	4.85
26	3.81	5.63	54	3.210	4.84
27	3.75	5.56	55	3.205	4.83
28	3.70	5.49	56	3.200	4.82
29	3.65	5.43	57	3.195	4.81
30	3.60	5.36	58	3.190	4.80
31	3.56	5.31	59	3.185	4.795
32	3.52	5.27	60	3.180	4.790
33	3.49	5.22	61	3.175	4.780
34	3.47	5.19	62	3.170	4.775
35	3.45	5.16	63	3.165	4.770
36	3.43	5.13	64	3.160	4.760
37	3.41	5.10	65	3.150	4.750

The use of the areometer and the tables belonging thereto for testing caustic lime presupposes the complete slacking of the caustic lime and settling of the lumps and grains of sand floating in the liquid. It is self-evident that a table which indicates the quantity of caustic lime corresponding to the separate degrees of an areometer does not possess the same degree of accuracy demanded from a table giving the content of soda, potash, common salt, etc.

*Table of content of caustic lime in milk of lime, according to Edward Mategcek.*

Degrees, Baumé.	Per cent. calcium oxide, CaO.	100 liters contain kilogr. CaO.	Degrees, Baumé.	Per cent. calcium oxide, CaO.	100 liters contain kilogr. CaO.
10	10.60	13.3	38	19.72	29.5
11	11.12	14.2	39	19.80	29.6
12	11.65	15.2	40	19.88	29.8
13	12.16	16.1	41	19.95	29.9
14	12.68	17.0	42	20.03	30.1
15	13.20	18.0	43	20.10	30.2
16	13.72	18.9	44	20.16	30.3
17	14.25	19.8	45	20.22	30.4
18	14.77	20.7	46	20.27	30.5
19	15.23	21.6	47	20.32	30.6
20	15.68	22.4	48	20.37	30.7
21	16.10	23.3	49	20.43	30.7
22	16.52	24.0	50	20.48	30.8
23	16.90	24.7	51	20.53	30.9
24	17.23	25.3	52	20.57	31.0
25	17.52	25.8	53	20.62	31.1
26	17.78	26.3	54	20.66	31.1
27	18.04	26.7	55	20.70	31.2
28	18.26	27.0	56	20.74	31.3
29	18.46	27.4	57	20.78	31.3
30	18.67	27.7	58	20.82	31.4
31	18.86	27.9	59	20.85	31.4
32	19.02	28.2	60	20.89	31.5
33	19.17	28.4	61	20.93	31.5
34	19.31	28.7	62	20.97	31.6
35	19.43	28.9	63	21.00	31.6
36	19.53	29.1	64	21.03	31.7
37	19.63	29.3	65	21.05	31.7

It has been previously mentioned (p. 223) that to causticize soda or potash are theoretically required,

For 100 parts potash . . . 40.6 parts of lime, or  
 " 1 part " . . . 0.406 " " (40.58)  
 For 100 parts calcined soda . . . 53.0 parts of lime, or  
 " 1 part " " . . . 0.53 " " (52.83).

So that with pure caustic lime, 100 parts of a

90 per cent. of potash require  $90 \times 0.406 = 36.54$  parts of lime.  
 75 " " "  $75 \times 0.406 = 30.45$  " "  
 80 " of soda "  $80 \times 0.53 = 42.40$  " "  
 60 " " "  $60 \times 0.53 = 31.80$  " "

The caustic lime is, however, never pure, and the wanting quantity of pure caustic lime has to be supplemented. Suppose

the alkalimetric test, in connection with the determination of the carbonic acid, shows a 95 per cent., 93 per cent., or 86 per cent. caustic lime, a sufficient quantity of more lime to make up the per cents. lacking of 100 has to be taken. The calculation is a simple equation and for the above examples as follows:—

*For soda:—*

$$\frac{100 \times 0.53}{95} = X; X = 55.79 \text{ lime.}$$

$$\frac{100 \times 0.53}{93} = X; X = 58.05 \text{ lime.}$$

$$\frac{100 \times 0.53}{86} = X; X = 61.63 \text{ lime.}$$

*For potash:—*

$$\frac{100 \times 0.406}{95} = X; X = 42.95 \text{ lime.}$$

$$\frac{100 \times 0.406}{93} = X; X = 43.65 \text{ lime.}$$

$$\frac{100 \times 0.406}{86} = X; X = 47.45 \text{ lime.}$$

The same calculation takes place to causticize a soda of  $n$  degrees or potash of  $n$  per cent. with a caustic lime of  $n$  per cent., for instance, 95 or 75 per cent.

$$\frac{95 \times 0.53}{95} = X; X = 53.00 \text{ lime.}$$

$$\frac{80 \times 0.53}{75} = X; X = 56.53 \text{ lime.}$$

$$\frac{95 \times 0.406}{95} = X; X = 40.60 \text{ lime.}$$

$$\frac{80 \times 0.406}{75} = X; X = 43.30 \text{ lime.}$$

An excess of lime should be avoided, the insoluble mass being thereby unnecessarily augmented, and, besides, the slimy calcium



hydrate does not settle as well as the granular calcium carbonate, which can throw down only a determined quantity of caustic lime as so-called calcium hydrocarbonate (see page 223).

The lime found in practice contains on an average only 90 per cent. of caustic lime, so that, generally speaking, the factor 0.583 may be taken for soda calculations and 0.451 for potash calculations, and for cold lyes even 0.63 and 0.50. The following tables have been calculated for the practice :—

*Table for soda and lime calculations.*

100 parts of n degree soda, $\text{Na}_2\text{CO}_3$ .	require the following parts of a lime of m per cent.								
	90	85	80	75	70	65	60	55	50
100	58.3	62.2	66.2	70.5	75.5	81.3	88.0	96.0	105.6
95	55.7	59.0	62.9	66.9	71.7	77.2	83.6	91.2	100.3
90	52.8	55.9	59.4	63.4	67.9	73.2	79.2	86.5	95.1
85	49.5	52.8	56.1	59.9	64.1	69.1	77.8	81.6	89.8
80	46.7	49.7	52.8	56.3	60.4	65.8	70.4	76.8	84.5
75	44.0	46.6	49.5	52.8	56.6	60.9	66.0	72.0	79.2
70	41.0	43.5	46.2	49.3	52.8	56.9	61.6	67.2	73.9
65	38.1	40.4	42.9	45.8	49.0	52.8	57.2	62.4	68.7
60	35.2	37.2	39.6	42.3	45.3	48.8	52.8	57.2	63.4
55	32.3	34.3	36.3	38.7	41.5	44.7	44.4	52.8	58.1
50	29.3	31.0	33.0	35.2	37.7	40.6	44.0	48.0	52.8

*Table for potash and lime calculations.*

100 parts of potash of n per cent. $\text{K}_2\text{CO}_3$ .	require the following parts of a lime of m per cent.								
	90	85	80	75	70	65	60	55	50
100	45.0	47.7	50.7	54.0	58.0	62.4	67.7	73.8	81.2
95	42.8	45.4	48.2	51.4	55.1	59.3	64.3	70.1	77.1
90	40.6	43.0	45.6	48.9	52.2	56.2	60.9	66.4	73.0
85	38.3	40.9	43.1	46.0	49.3	53.1	57.5	62.7	69.0
80	36.1	38.2	40.6	43.3	46.4	50.0	54.1	59.0	65.0
75	33.8	35.8	38.0	40.6	43.5	46.8	50.7	55.3	60.9
70	31.6	33.4	35.5	37.9	40.6	43.7	47.3	51.6	56.8
65	29.3	31.0	33.0	35.2	37.7	40.6	44.0	48.0	52.7
60	27.1	28.6	30.5	32.5	34.8	37.5	40.6	44.3	48.7
55	24.8	26.3	27.9	29.7	31.9	34.4	37.2	40.6	44.6
50	22.5	23.8	25.3	27.0	29.0	31.3	33.8	36.9	40.6

To causticize a solution of soda or potash of unknown strength, determine the degrees with Baumé's or Twaddle's areometer, and

from the quantity of fluid calculate, with the assistance of the tables on page 232, the content of soda or potash; the quantity of lime required for causticizing is to be determined at the same time.

Suppose, for example, we have 600 litres of potash solution showing  $30^{\circ}$  B., then, according to the table, page 232, giving the content of soda and potash solutions in kilogrammes,  $6 \times 43.6 = 261.6$  kilogrammes. To causticize 100 kilogrammes 45 kilogrammes of lime are required; therefore, for 261.6 kilogrammes of potash  $\frac{261.6 \times 45}{100}$  or  $261.6 \times 0.45 = 97.47$  kilogrammes

of lime, and these yield, according to the table, page 234, giving the content of lime in kilogrammes, 400 litres of milk of lime of  $22\frac{1}{2}^{\circ}$  B.,  $4 \times 24.35 = 97.40$  of lime.

The following tables show the content of effective alkali in the lyes according to degrees B. :—

*Table showing the percentage of oxides and hydroxides in caustic soda and caustic potash lyes, with the corresponding specific gravities and areometer degrees of Baumé and Twaddle at 59° F.*

Degrees, Baumé.	Degrees, Twaddle.	Specific gravity.	Caustic soda lye.		Caustic potash lye.	
			Per cent. by weight, sodium oxide.	Per cent. by weight, sodium hydroxide.	Per cent. by weight, potassium oxide.	Per cent. by weight, potassium hydroxide.
0.0	0.0	1.0000	—	—	—	—
0.5	0.7	1.0035	0.23	0.30	0.35	0.45
1.0	1.4	1.0070	0.47	0.61	0.70	0.90
1.5	2.1	1.0105	0.70	0.90	1.05	1.30
2.0	2.8	1.0141	0.93	1.20	1.40	1.70
2.5	3.6	1.0177	1.26	1.60	1.80	2.15
3.0	4.4	1.0213	1.55	2.00	2.20	2.60
3.5	5.1	1.0249	1.83	2.36	2.55	3.05
4.0	5.8	1.0286	2.10	2.71	2.90	3.50
4.5	6.6	1.0323	2.35	3.03	3.35	4.00
5.0	7.4	1.0360	2.60	3.35	3.80	4.50
5.5	8.2	1.0397	2.85	3.67	4.25	5.05
6.0	9.0	1.0435	3.10	4.00	4.70	5.60
6.5	9.7	1.0473	3.35	4.32	5.05	6.00
7.0	10.4	1.0511	3.60	4.64	5.40	6.40
7.5	11.2	1.0549	3.85	4.96	5.80	6.80
8.0	12.0	1.0588	4.10	5.29	6.20	7.40
8.5	12.7	1.0627	4.32	5.58	6.55	7.80
9.0	13.4	1.0667	4.55	5.87	6.90	8.20
9.5	14.2	1.0706	4.82	6.21	7.30	8.70
10.0	15.0	1.0746	5.08	6.55	7.70	9.20
10.5	15.8	1.0787	5.37	6.76	8.10	9.65
11.0	16.6	1.0827	5.67	7.31	8.50	10.10
11.5	17.3	1.0868	5.84	7.66	8.85	10.50
12.0	18.2	1.0909	6.20	8.00	9.20	10.90
12.5	19.1	1.0951	6.46	8.34	9.65	11.45
13.0	20.0	1.0992	6.73	8.68	10.10	12.00
13.5	20.8	1.1035	7.06	9.05	10.45	12.45
14.0	21.6	1.1077	7.30	9.42	10.80	12.90
14.5	22.4	1.1120	7.55	9.74	11.25	13.35
15.0	23.2	1.1163	7.80	10.06	11.60	13.80
15.5	24.1	1.1206	8.15	10.51	12.00	14.30
16.0	25.0	1.1250	8.50	10.97	12.40	14.80
16.5	25.9	1.1294	8.84	11.42	12.80	15.25
17.0	26.8	1.1339	9.18	11.84	13.20	15.70
17.5	27.6	1.1383	9.49	12.24	13.55	16.10
18.0	28.4	1.1423	9.80	12.64	13.90	16.50
18.5	29.4	1.1474	10.15	13.00	14.35	17.15
19.0	30.4	1.1520	10.50	13.55	14.80	17.60
19.5	31.4	1.1566	10.82	13.86	15.20	18.10
20.0	32.4	1.1613	11.14	14.37	15.60	18.60
20.5	33.6	1.1660	11.43	14.75	16.00	19.05
21.0	34.2	1.1707	11.73	15.13	16.40	19.50
21.5	35.1	1.1755	12.03	15.50	16.80	20.00
22.0	36.0	1.1803	12.33	15.91	17.20	20.50
22.5	37.0	1.1852	12.66	16.38	17.60	20.95
23.0	38.0	1.1901	13.00	16.77	18.00	21.40
23.5	39.0	1.1950	13.35	17.22	18.40	21.90
24.0	40.0	1.2000	13.70	17.67	18.80	22.50
24.5	41.0	1.2050	14.05	18.12	19.20	22.85



*Table showing the percentage of oxides and hydroxides in caustic soda and caustic potash lyes.—Continued.*

Degrees, Baumé.	Degrees, Twaddle.	Specific gravity.	Caustic soda lye.		Caustic potash lye.	
			Per cent. by weight, sodium oxide.	Per cent. by weight, sodium hydroxide.	Per cent. by weight, potassium oxide.	Per cent. by weight, potassium hydroxide.
25.0	42.0	1.2101	14.40	18.58	19.60	23.30
25.5	43.0	1.2152	14.74	19.08	19.95	23.75
26.0	44.0	1.2202	15.18	19.58	20.30	24.20
26.5	45.1	1.2255	15.57	20.08	20.70	24.65
27.0	46.2	1.2308	15.96	20.59	21.10	25.10
27.5	47.1	1.2361	16.36	21.00	21.50	25.60
28.0	48.2	1.2414	16.76	21.42	21.90	26.10
28.5	49.3	1.2468	17.18	22.03	22.30	26.50
29.0	50.4	1.2522	17.55	22.64	22.70	27.00
29.5	51.5	1.2576	17.85	23.15	23.10	27.50
30.0	52.6	1.2632	18.35	23.67	23.50	28.00
30.5	53.7	1.2687	18.78	24.24	23.85	28.45
31.0	54.8	1.2743	19.23	24.81	24.20	28.90
31.5	55.9	1.2800	19.61	25.30	24.60	29.35
32.0	57.0	1.2857	20.00	25.80	25.00	29.80
32.5	58.2	1.2905	20.40	26.31	25.40	30.25
33.0	59.4	1.2973	20.80	26.83	25.80	30.70
33.5	60.5	1.3032	21.02	27.31	26.25	31.25
34.0	61.6	1.3091	21.55	27.80	26.70	31.80
34.5	62.8	1.3151	21.95	28.31	27.10	32.25
35.0	64.0	1.3211	22.35	28.83	27.50	32.70
35.5	65.2	1.3272	22.67	29.38	27.90	33.20
36.0	66.4	1.3333	23.20	29.93	28.30	33.70
36.5	67.7	1.3395	23.75	30.57	28.80	34.30
37.0	69.0	1.3458	24.20	31.22	29.30	34.90
37.5	70.2	1.3521	24.68	31.85	29.75	35.40
38.0	71.4	1.3585	25.17	32.47	30.20	35.90
38.5	72.7	1.3649	25.68	33.08	30.60	36.40
39.0	74.0	1.3714	26.12	33.69	31.00	36.90
39.5	75.3	1.3780	26.61	34.38	31.40	37.35
40.0	76.6	1.3846	27.10	34.96	31.80	37.80
40.5	78.5	1.3913	27.60	35.65	32.25	38.35
41.0	79.4	1.3981	28.10	36.25	32.70	38.90
41.5	80.7	1.4049	28.58	36.86	33.10	39.40
42.0	82.0	1.4118	29.05	37.47	33.50	39.90
42.5	83.4	1.4187	29.56	38.13	33.95	40.40
43.0	84.8	1.4267	30.08	38.80	34.40	40.90
43.5	86.2	1.4328	30.54	39.39	34.90	41.50
44.0	87.6	1.4400	31.00	39.99	35.40	42.10
44.5	89.0	1.4472	31.50	40.75	35.95	42.75
45.0	90.6	1.4545	32.10	41.41	36.50	43.40
45.5	92.2	1.4619	32.65	42.12	37.00	44.00
46.0	93.6	1.4694	33.20	42.83	37.50	44.60
46.5	95.1	1.4769	33.80	43.66	38.00	45.20
47.0	96.6	1.4845	34.40	44.38	38.50	45.80
47.5	98.1	1.4922	35.05	45.27	39.05	46.45
48.0	99.6	1.5000	35.70	46.15	39.60	47.10
48.5	101.2	1.5079	36.30	46.87	40.15	47.70
49.0	102.8	1.5158	36.90	47.60	40.60	48.30
49.5	104.4	1.5238	37.45	48.81	41.05	48.85
50.0	106.0	1.5319	38.00	49.02	41.50	49.40

*Table giving the content of soda and potash lye in kilogrammes according to degrees Baumé at 59° F.*

Degrees, Baumé.	100 liters contain, kilogrammes.		100 liters contain, kilogrammes.	
	Sodium oxide, Na <sub>2</sub> O.	Sodium hydroxide, NaHO.	Potassium oxide, K <sub>2</sub> O.	Potassium hydroxide, KHO.
1	0.4	0.6	0.7	0.9
2	0.9	1.2	1.4	1.7
3	1.6	2.1	2.2	2.6
4	2.2	2.8	3.0	3.6
5	2.7	3.5	3.9	4.6
6	3.2	4.2	4.9	5.8
7	3.8	4.9	5.7	6.7
8	4.3	5.6	6.6	7.8
9	4.9	6.3	7.4	8.8
10	5.5	7.0	8.3	9.9
11	6.1	7.9	9.2	10.9
12	6.8	8.7	10.0	11.9
13	7.4	9.5	11.1	13.2
14	8.1	10.4	11.9	14.3
15	8.7	11.2	12.9	15.3
16	9.6	12.3	14.0	16.7
17	10.4	13.4	15.0	17.8
18	11.2	14.4	15.9	18.8
19	12.1	15.6	17.0	20.3
20	12.9	16.7	18.1	21.6
21	13.7	17.7	19.2	22.8
22	14.6	18.8	20.3	24.2
23	15.5	20.0	21.4	25.5
24	16.4	21.2	22.6	26.9
25	17.4	22.5	23.7	28.2
26	18.5	23.9	24.8	29.5
27	19.6	25.3	26.0	30.9
28	20.8	26.6	27.2	32.4
29	22.0	28.3	28.4	33.8
30	23.0	29.9	29.7	35.3
31	24.5	31.6	30.8	36.8
32	25.7	33.2	32.1	38.5
33	27.0	34.8	33.8	39.8
34	28.2	36.4	34.9	40.6
35	29.5	38.1	36.3	43.2
36	30.9	39.9	37.7	44.9
37	32.6	42.0	39.4	46.9
38	34.2	44.1	41.0	48.7
39	35.9	46.2	42.5	50.6
40	37.5	48.3	44.0	52.2
41	39.2	50.6	45.7	54.3
42	40.0	52.8	47.2	56.3
43	42.8	55.3	49.0	58.2
44	44.6	57.5	50.9	60.5
45	46.6	60.2	53.0	63.1
46	48.7	62.9	54.9	65.5
47	51.0	65.8	57.1	67.9
48	53.5	69.1	59.3	70.6
49	55.9	72.1	61.5	73.1
50	58.1	75.0	62.5	75.6

The correct proportions of the quantity of caustic alkali and alkaline carbonate in the lyes being of great importance for the success of the soaps, it is recommended to the soap-boiler not to depend on the determination by Baumé's or Twaddle's areometer alone, but to test the lyes as to their content of caustic alkalies and alkaline carbonates. By a simple titration of the lyes the entire content of caustic alkali and alkaline carbonate can be determined according to the calculation either as caustic alkali or alkaline carbonate. The content of each can, however, be also readily determined by the alkalimetric method, by first ascertaining the total content of alkali in a sample, and separating from a second sample the alkaline carbonate, with the assistance of barium chloride solution,\* and determining the quantity of caustic alkali in the fluid filtered off from the precipitate. The difference between the first and second determinations gives the quantity of non-causticized alkali.

To test, for instance, a strong soda lye, weigh off 40 grammes in a beaker-glass, pour them in a liter flask, rinse the beaker-glass with water, pour the latter into the flask, and fill the flask with water to the liter mark. The one liter of liquid contains now 40 grammes of lye, 100 cubic centimetres, therefore 4 grammes. Bring, by means of a pipette, 100 cubic centimetres from the liter flask into a beaker-glass, color blue with litmus tincture, and add by means of a burette sufficient normal acid so that the liquid remains entirely red after boiling. Suppose now 21.5 cubic centimetres of normal acid had been used. To determine the quantity of acid added in excess, add carefully, normal alkali solution until the liquid acquires a perceptibly blue color. Suppose the quantity of normal alkali solution used is 1.5 cubic centimetres, then  $21.5 - 1.5$ , therefore 20 cubic centimetres of normal acid have been used. Now as 1 cubic centimetre of normal acid corresponds to 1 per cent. of caustic soda, the lye contains 20 per cent. of caustic soda, provided all the alkali present is caustic soda.

Now take by means of a pipette 100 cubic centimetres more

\* The barium chloride forms with the alkaline carbonate barium carbonate, which is insoluble in water, and soluble alkaline chlorides, which are not affected by the test acid.



from the liter flask, bring them into a beaker-glass and add barium chloride until a precipitate is no longer formed. Allow the precipitate to settle, then filter through a paper filter in a glass-funnel, and wash the residue upon the filter with distilled water until the liquid running off from the funnel no longer colors red litmus paper blue. Now color the filtrate blue with litmus tincture, and titrate with normal acid. No carbonic acid being present to endanger the appearance of the color, boiling of the liquid is not required. Suppose for the second titration 16 cubic centimetres of normal acid had been used, then the lye contains 16 per cent. of caustic soda. The difference, 4 per cent., between the first and second titration gives the quantity of non-causticized soda. This, being present as carbonate, was in the first titration calculated as caustic soda, and we must, therefore, recalculate according to the equation,  $40 : 53 = 4 : x = 5.3$ . Hence the lye contains 16 per cent. of caustic soda and 5.3 per cent. of sodium carbonate.

The method of testing potash lye is the same, except that 56 grammes of lye are weighed off and increased to 1 liter. Determine first the total content of alkali in 100 cubic centimetres, and the caustic potash in another 100 cubic centimetres after separating the potassium carbonate with barium chloride. The potassium carbonate is calculated from the difference between the first and second titration, taking, however, into consideration that caustic potash is to potassium carbonate as 56 : 69.

## CHAPTER X.

## MACHINES AND UTENSILS FOR THE MANUFACTURE OF SOAP.

*Kettles.*—Kettles, called in England soap-pans or coppers, are vessels in which by means of heat the soap-boiler combines fatty bodies with lyes of potash or soda to form soap. Their dimensions vary according to requirement, an allowance of 14 cubic feet being generally made for each 220 pounds of fat to be saponified. The older style of kettle employed in smaller factories for boiling soap over an open fire consists of two parts, of which the lower one, the actual kettle, of cast-iron or copper, is set in masonry so that the heat acts upon the bottom. The upper portion, the so-called curb, is a hollow cone of iron-bound staves made to fit the flange of the lower portion, and can be made to extend as high as desired. The cone stands erect, but should be strongly and tightly fastened and joined to the lower portion. In this way a kettle may be enlarged at much less cost than for a kettle wholly of iron. Should there be several kettles, they are placed on a line with each other and over a furnace beneath. To each kettle a pipe of about two inches diameter is adapted which serves for the discharge of the sub-lye.

The more modern kettles are of the shape shown in Fig. 29, and generally constructed of wrought-iron plates riveted together. Some kettles have flat bottoms, others have convex or concave bottoms. Experience has shown the latter arrangement to be the best and most convenient for use. With an open fire the heating gases are generally allowed to pass under the bottom of the kettle and from there are conducted upward and around the kettle, and finally into the chimney flue. With the high temperature of the heating gases formed by the combustion of the coal, the bottom of the kettle suffers considerably if placed immediately over the grate, and scorching of the soap can only be prevented by very

careful handling of the fire and constant stirring. This evil is overcome by the manner of bricking in the kettles as shown in

Fig. 29.

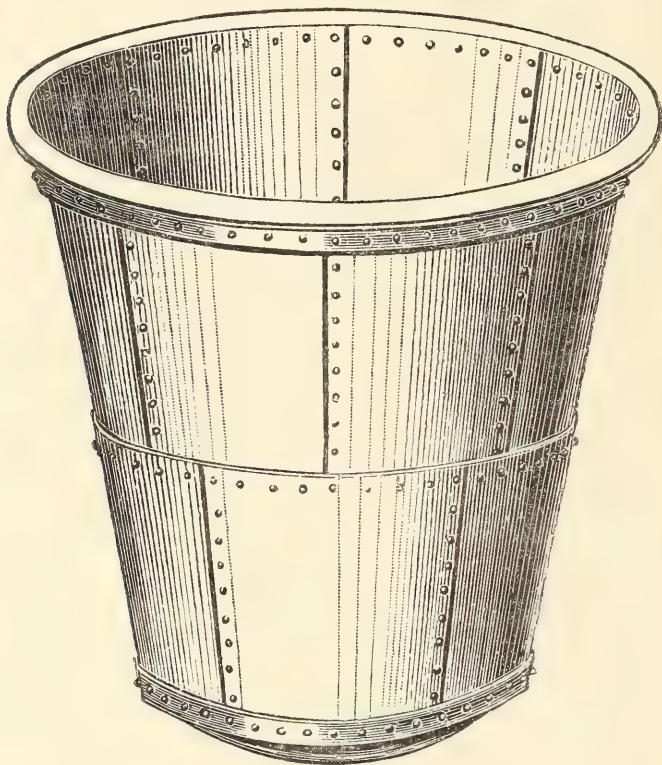


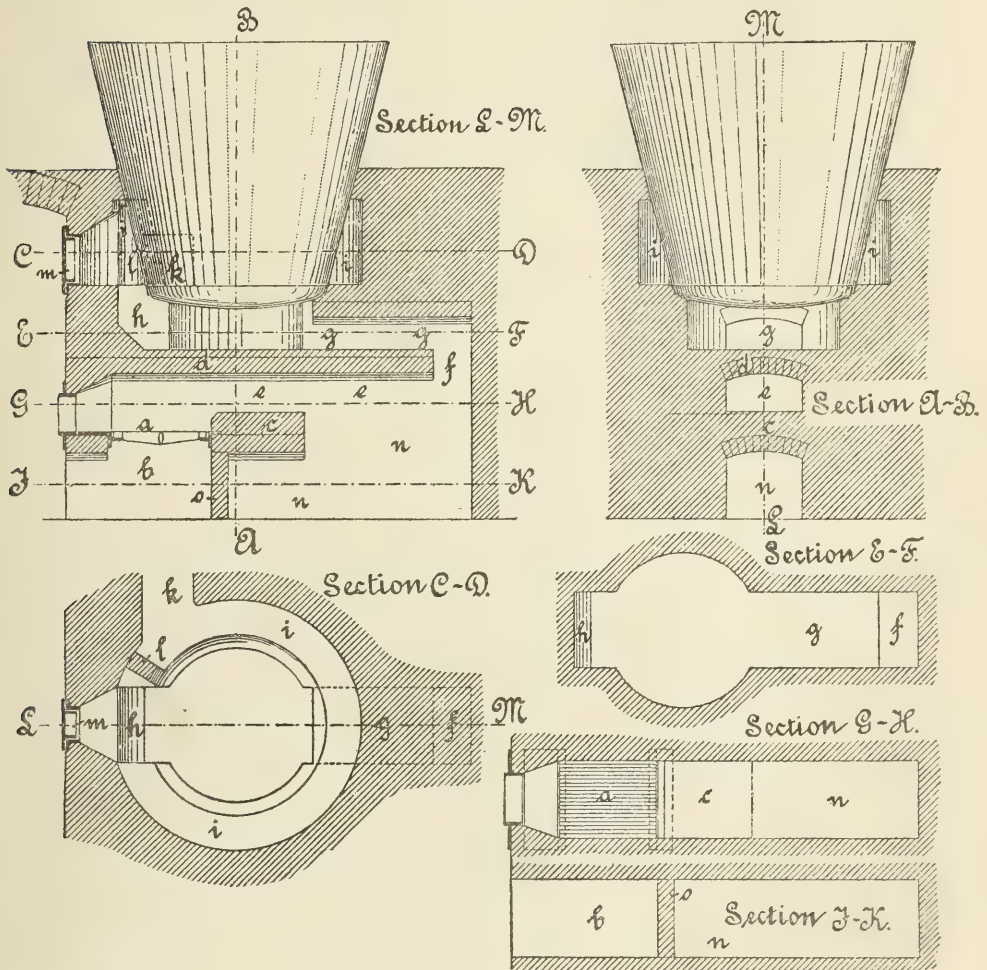
Fig. 30. The characteristic feature of this arrangement is a protecting arch placed between the bottom of the kettle and the grate, so that the flame, before it reaches the bottom of the kettle, yields up its strongest heat by passing under this arch.

The heating gases developed upon the grate *a*, below which is the ash-pit *b*, pass over the fire-bridge *c* into the channel *e*, which is covered by the fire-proof arch *d*; on the end of the channel *e*, the gases ascend upward into the vertical channel *f*, and finally reach the bottom of the kettle through the flue *g*. After passing under the bottom of the kettle they ascend upward through the inclined channel *h* into the lateral flue *i* running entirely around the kettle, and at *k* pass into the chimney flue. The circulation of the gases around the kettle is effected by the brick partition *l* built in the flue *i*. *m* is a hermetically closed manhole for cleaning the flues. The space *n*, located nearly behind the fire-bridge



*c*, serves for the reception of flying ashes. It is closed at *o* by brickwork.

Fig. 30.

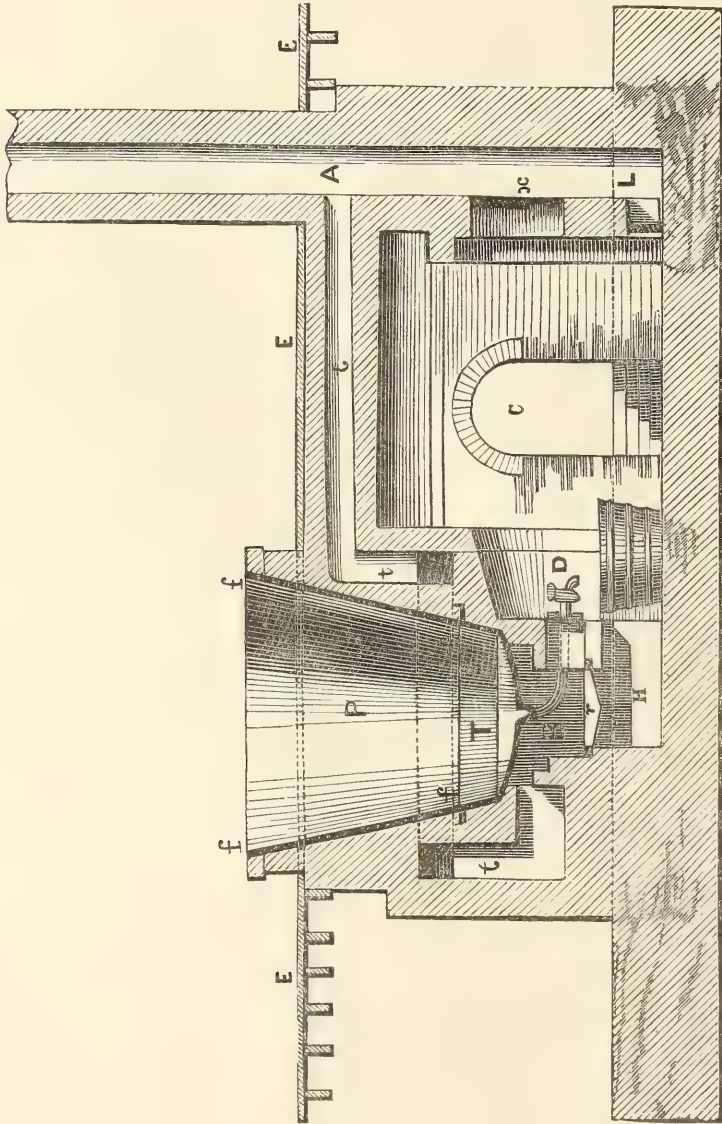


The above arrangement, which has proved very satisfactory in practice, permits the adjustment of any desired special contrivances, as, for instance, the smokeless combustion of the coal, discharge-cocks for the sub-lye and soap, etc.

A kettle provided with a curb and heated over an open fire is shown in Fig. 31. The kettle is constructed of wrought-iron plates, and the curb *ffff*, which expands into a cone to afford space for the soap to rise, of brick thoroughly cemented. The fire-place *B* is separated from the ash-pit *H* by the grate *r*. The fire, after striking the bottom of the kettle, passes through the

flue *tt*, half around the side of the kettle, into the chimney *A*. This is accessible for the purpose of cleaning by the manhole *x*; the soot collected is thrown into the pit *L*. A wrought-iron

Fig. 31.



pipe *D*, provided with a cock, leads from the lowest part of the kettle for the discharge of the spent lye, which is caught in a vessel placed under the cock.

*Heating the kettles by steam* offers many advantages: the curb of the kettle need not be so high, since the process can be better

regulated by the admission of more or less steam; the kettles suffer less damage, all scorching is avoided, saponification is more quickly and completely effected, and purer and whiter soaps are obtained. We would, however, remark that the steam serves only for the actual formation of the soap; the actual boiling to the so-called curd or grain, whereby an evaporation of water takes place, can only be effected over an open fire.

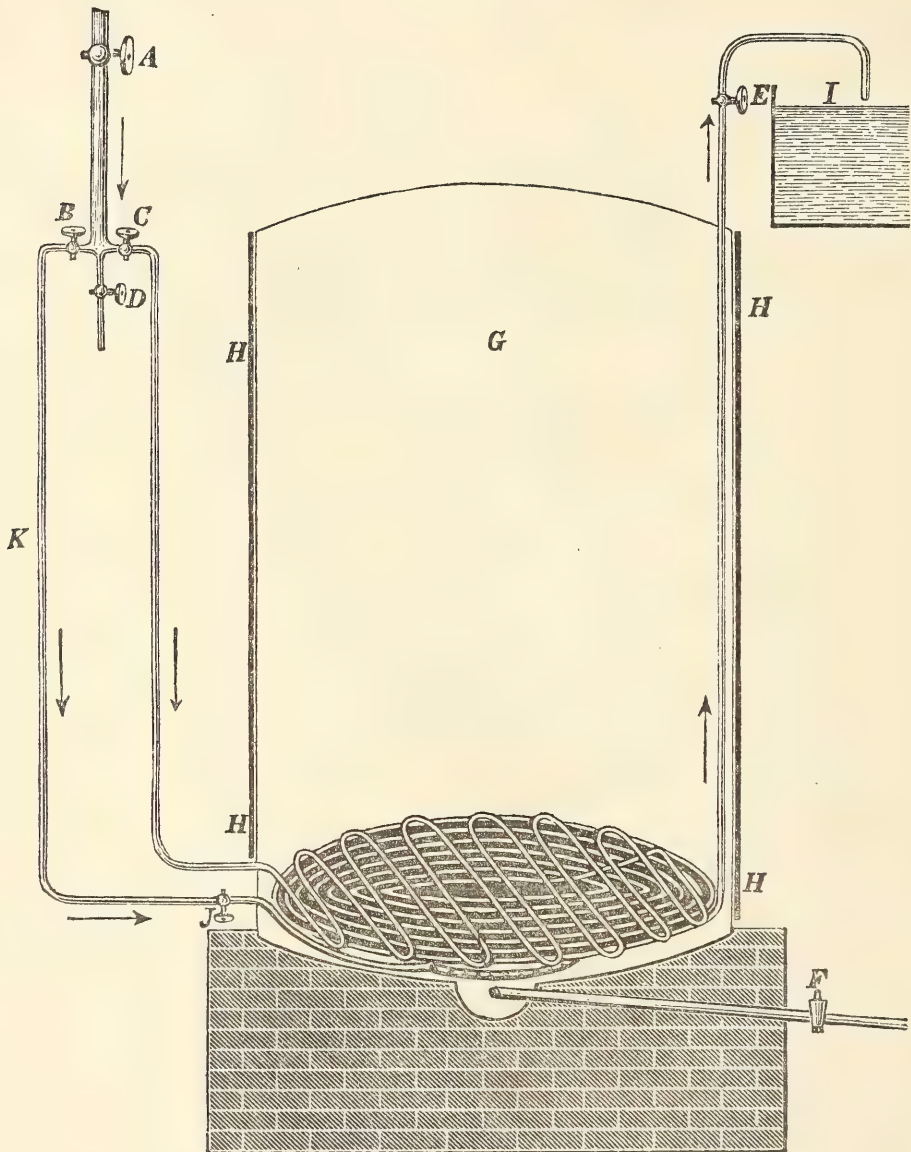
The first manufacturers who used steam discharged it directly into the mass of soap; the result was that the water produced by the condensation of the steam considerably weakened the lyes used to saponify the fat. The next idea was to cause the steam to circulate in the kettle, within a double casing, in such a manner that the water produced by the condensation of the steam should not mix with and weaken the lyes. This system is still in use in some establishments, but it has the inconvenience that the sides of the kettle are heated too much and the bottom not enough, the result being that ebullition never proceeds very regularly and is more pronounced on the sides than in the centre. In new factories the soap is, at the present time, directly heated by means of steam circulating in a wrought-iron coil placed about 3 to 4 inches above the bottom. Fig. 32 shows such an arrangement with direct and indirect steam. *G* is the kettle of wrought-iron plates; *H H H H* a wooden jacket about  $1\frac{3}{4}$  inches thick, to prevent too rapid cooling off in winter. *A* is a stop-cock on the main steam-pipe; *B* and *C* stop-cocks on the pipe system connected with the direct and indirect coils. *D* is a safety-cock, *E* cock for regulating the water condensing in the steam-coil, *I* lye reservoir, *F* discharge-pipe for the sub-lye. The stopcock *J* prevents the lye or soap from rising back into the steam-pipe *K* after the operation is finished. The size of the pipes depends on that of the kettle; generally they have, however, a clear diameter of  $1\frac{1}{2}$  inches. The cocks *A* and *D* are provided for the sake of safety, so that in case the cocks *B* and *C* are not absolutely tight, the steam can nevertheless be prevented from entering the kettle. After finishing each boiling, the cocks *A*, *B*, and *C* are closed and the cock *D* is opened. In case the cock *A* is not tight, the steam escapes from *D* without fear of danger.

Kettles of colossal size, capable of turning out 50 tons of fin-



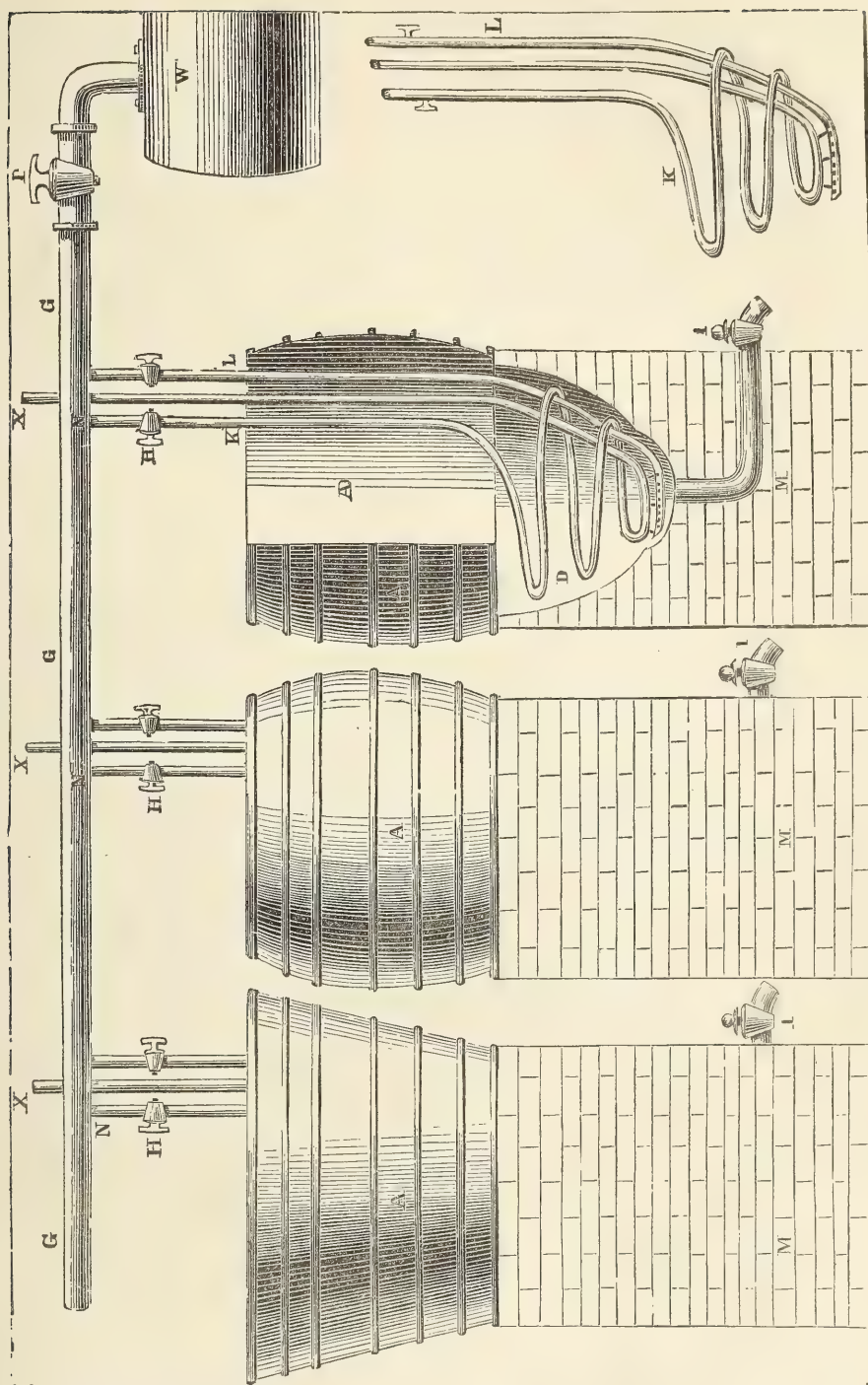
ished soap at one operation, are by no means uncommon, and some of the large factories in this country have built even still

Fig. 32.



larger kettles, requiring a building of three stories to contain them. Kettles whose contents are boiled by steam may be of any desired shape, circular, oval, or rectangular, provided that the steam-pipes be carried into the corners (if any) and be so arranged as to insure uniformity of ebullition throughout the whole mass.

Fig. 33.



There is no necessary proportion between diameter and depth ;\* in England the diameter is to depth as 1 to 1, or 1 to 1.25, or 1

\* A kettle 15 feet in diameter and 15 feet deep will turn out 25 to 30 tons of soap.

to 1.5, while in this country the ratio is increased as far as 1 to 2, 1 to 2.5, and 1 to 3.

Fig. 33 shows a steam series arranged for three kettles, each being used only for a certain kind of soap. *G* is the main pipe or feeder, which is attached to the steam-boiler, *W*, of the establishment. It is stationary and generally fitted against the wall immediately above the kettles. The latter are partly of iron and partly of wood, the upper portion or curb *A* being of wood, well hooped round by iron rings, and the lower portion, *D*, of cast-iron, and so shaped that the worm may hug closely to the sides without loss of room and the "blow-pipe" fit snugly to the bottom. For the convenience of drawing off the spent lye there are attached a pipe and cock *I*. Each of the kettles, resting upon a hollow pile of masonry, *M*, is furnished with a welded wrought-iron worm, which connects with the main feeder at *N*, and serves as the boiling medium of the soap paste. The steam is let on or off by opening or shutting the cock *H*, and the waste steam is conducted through the other end of the worm *X*, which passes upward by the side of its inlet and thence out in any convenient way through the wall of the factory. Also affixed to the main feeder is another pipe with a stop-cock attached, and leading immediately downwards to the bottom of the kettle, where it is fitted to a circular iron tube, pierced around its circumference with holes. It is set immediately below the worm and is called the "blow-pipe," serving to give additional heat occasionally to the contents of the kettle, as well as to stir it up when necessary—an operation more effectually executed in this way than by a crutch in the hands of a workman. The whole interior arrangement of the boiling kettle is seen at the figure *A D*, the worm detached at *K* and the "blow-pipe" at *L*. These kettles are worked much in the same manner as the ordinary ones over an open fire, except that they require less attention.

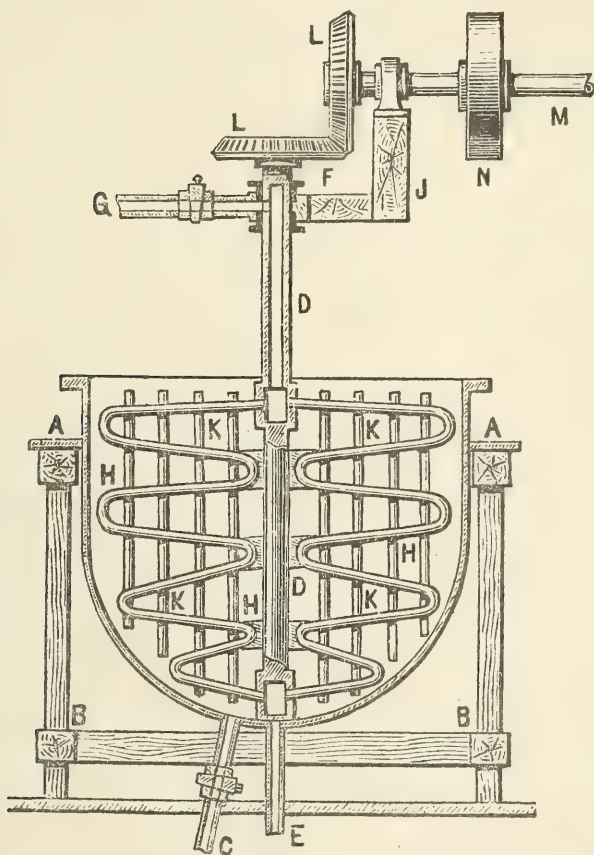
An apparatus for heating with steam, combined with a mechanical motion for stirring the liquid in place of the ordinary hand-crutch, is shown in Fig. 34.

In the kettle *A* moves in the stuffing-box *F* a vertical shaft *D*, hollow both in its upper and lower parts but solid in the middle; it is set in motion by the shaft *M* with pulley *N* by means of the



set of gearings *L*. On the vertical shaft *D* the upper hollow part is connected with the lower hollow part by bent pipes *H*, which are fastened to the frame *K*. The lower part of the shaft

Fig. 34.



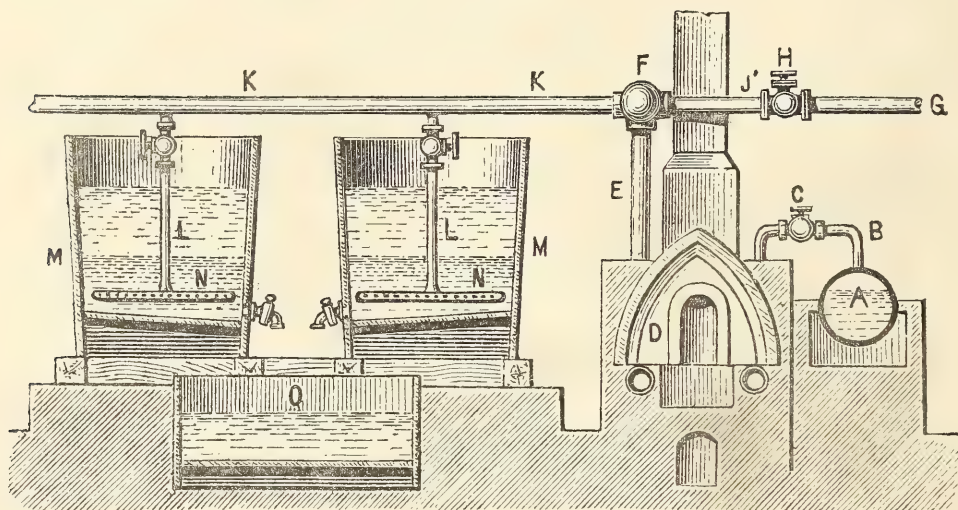
*D* runs through the bottom of the kettle *A*, fitting sufficiently tight to prevent the soap and lye from escaping, yet loose enough to be turned with ease. To heat the contents of the kettle, steam is admitted into the vertical shaft *D* and the serpentine pipes *H* connected with it by opening a cock on the steam-pipe *G*. The condensed water is drawn off at *E*, while the pipe *C*, which is provided with a cock, serves for the discharge of the spent lye.

By the intimate contact of the fat with the lye effected by the stirrer, saponification takes place in an extraordinarily short time.

A still more advantageous process is boiling the soap with superheated steam. Fig. 35 shows the arrangement of such an

apparatus. *A* is a steam-boiler of ordinary construction, *B* is a steam-pipe provided with a stop-cock *C*, and leads to the super-heater *D*. The pipe *E* conducts the superheated steam from the superheater *D* to the receiver *F*. Connected with the receiver *F* is a pipe *G*, which supplies air from a force-pump, the admission of air being regulated by the valve *H*. The air is conducted through the pipe *J* into the receiver *F*, where it mixes with the

Fig. 35.



superheated steam, and is then introduced into the apparatus. This is effected from the main feeder *K*, from which it is conducted by the pipes *L*, provided with stop-cocks, into the separate kettles. The pipes *L* are connected with the vertical pipes *N*, which are perforated on one side, and are revolved by the mixture of air and steam issuing from the perforations, thus effecting a stirring of the contents of the kettles. *O* is a tank for receiving the spent lye discharged into it by means of cocks.

The manner of conducting the operation is as follows: The lye and fats being introduced into the vats *M M*, superheated steam alone is introduced from the superheater *D* through the pipes and connecting pieces *E*, *F*, *K*, *L*, and the mass boiled. If now the mass is to be stirred, air is introduced into the apparatus by opening the valve *H*. It will be seen that the workman has perfect control of the operation, being able by simply turning the

cock *C* or *H* to increase or diminish the heat, or to stir or leave the contents of the vats *MM* at rest.

To prevent the contents of the kettle from rising and boiling over, as they are apt to do when saponification is taking place, and also later on after the steam is turned off, a little piece of machinery may be used. It consists essentially of a paddle-wheel formed of four paddles of strong wide hoop iron. Figs. 36 and 37 represent the apparatus. The shaft of the wheel rests

Fig. 36.

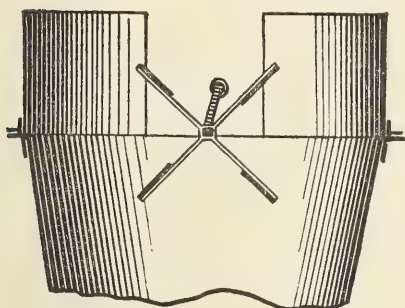
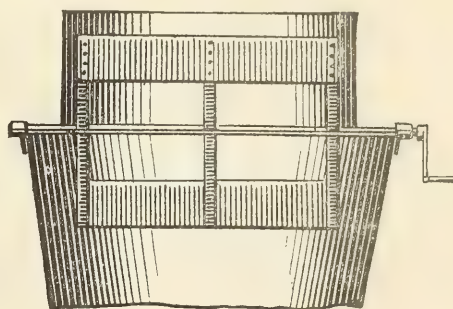


Fig. 37.



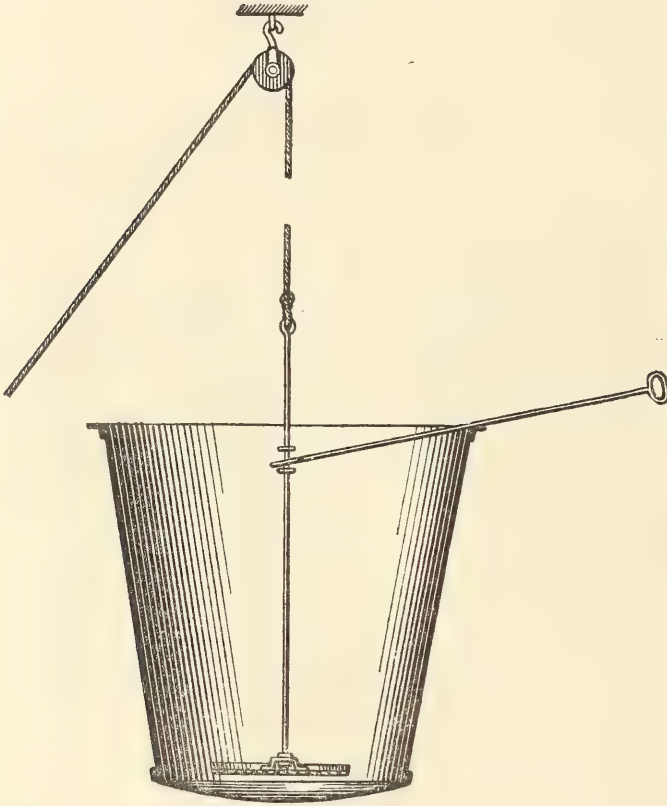
in a journal in the rim of the kettle, and is set in motion by a crank or a suitable gearing. On the rim of the kettle opposite the paddles are fastened a couple of sheet-iron plates to prevent the splashing over of drops of soap.

In most small factories the filling is crutched into the finished soap by means of hand-crutches. The following apparatus is recommended as a substitute. In the ceiling of the factory immediately over the centre of the kettle is fastened a strong wooden pulley over which runs a rope. To this rope is suspended the crutch of iron or wood, Fig. 38, the handle of which should reach from the bottom to the rim of the kettle, or somewhat above it. Somewhat below the rim of the kettle, about even with the ordinary level of the soap to be filled, is inserted in the handle of the crutch an iron or wooden guide-rod, with the assistance of which the crutch can be moved in every direction, and, if required, pressed down, though as a rule it falls down by its own weight, which, however, should not exceed 22 to 32 pounds. The lower portion of the crutch may be conveniently twice as large as that of an ordinary hand-crutch. One work-



man hoists the crutch up by means of the rope while another directs it.

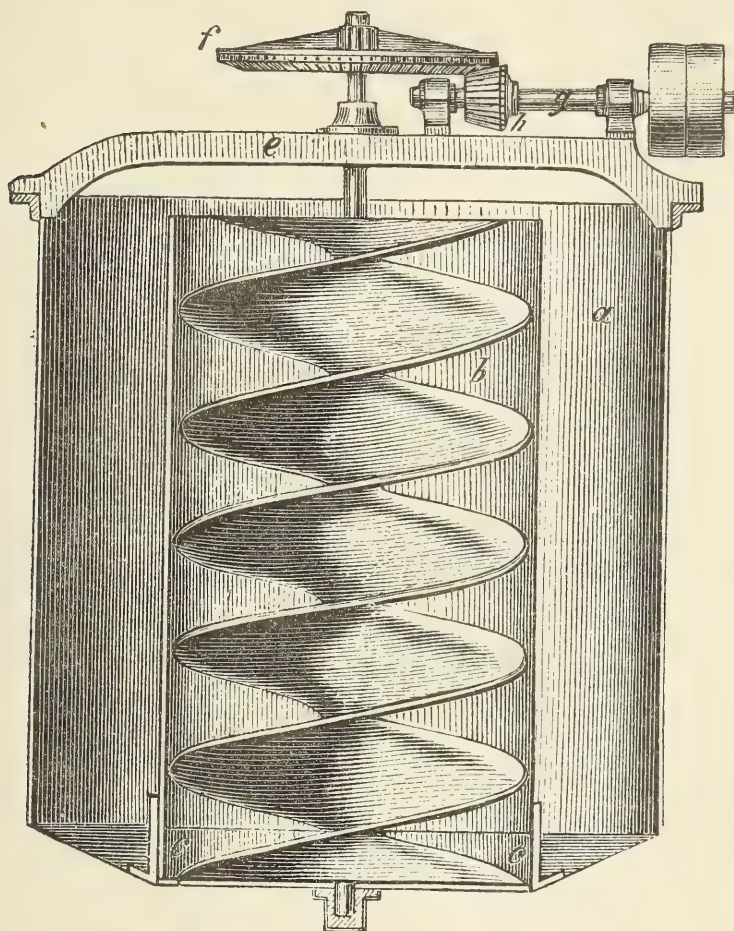
Fig. 38.



In this country, where the resin-grain soap is generally filled with soda solution, special mixing machines are in use. Fig. 39 shows such an apparatus constructed by A. Doll, of Cleveland, Ohio. It is constructed entirely of iron and provided with a safety-valve. In an iron cylinder *a*, about 4 feet in diameter and of the same height, and provided below with an aperture, which can be closed, is placed a second cylinder *b*, about  $2\frac{1}{2}$  feet in diameter, which, however, does not reach to the bottom but rests upon legs, *c*, 4 to 5 inches high. In this cylinder revolves an Archimedean screw *d*, the lower pivot of which runs in a step-bearing. The upper part ends, however, in a shaft, which runs in a journal placed in the transverse *e*, and bears the conical driving-wheel *f*, which catches into the smaller gearing *h* keyed upon the shaft *g*. When the apparatus is set in motion, the soap

is taken up by the screw passing close to the bottom and conveyed upward, where it falls into the outer cylinder, this being continued until in the course of about ten minutes it is intimately mixed.

Fig. 39.

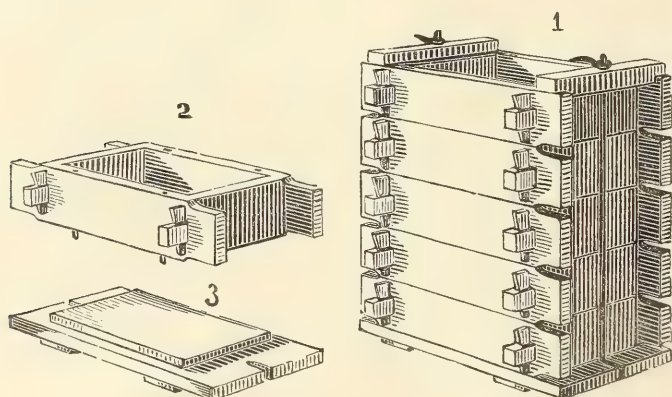


*Soap frames.*—This name is given to reservoirs of wood or iron, into which the soap is run when drawn from the kettle, in order that it may cool. They must be so arranged that they can be easily and conveniently taken apart and put together and perfectly tight so as not to permit any leakage of the warm liquid soap. They ordinarily have the form of a parallelogram; their dimensions vary according to the quantity of soap to be run into them, and are generally regulated by the capacity of the kettle.

*Frames of wood.*—These frames are made of oak or pine. Those of oak are costly and have the disadvantage of coloring

the soap ; the others do not present this inconvenience and are to be preferred. In this country wooden frames are generally constructed in four movable sections made of pine boards about two or three inches thick and lined with very thin sheet-iron. The sections should fit closely upon each other when piled in a series. The bottom of the frame may be made of wood or of brick ; in the case of mottled-curd soap it is convenient to have a well in the bottom to receive the lye which drains from the soap. When the soap is cold the wooden frames are lifted off, one at a time, and the soap remains upon the bottom ready to be divided into

Fig. 40.



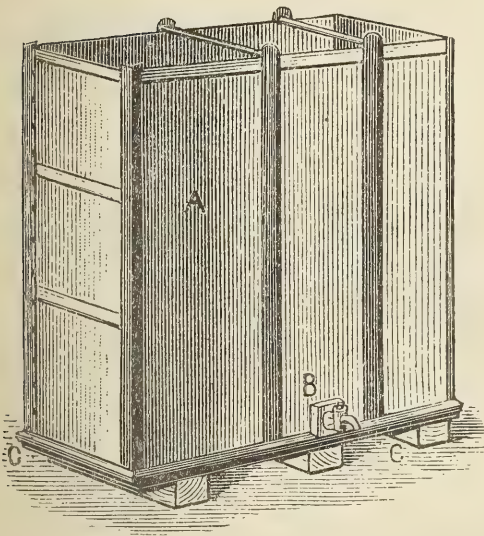
bars. Fig. 40 shows a frame of five sections ready for receiving the soap-paste. A single section is shown in Fig. 40 (2), and the bottom of the frame in Fig. 40 (3).

The German frames, like those of this country, are also constructed so that they may be readily taken apart, being set up by nuts and screws. Their floors are also movable. They consist of two layers of deal boards, in the upper of which are four grooves, fitting with the projections in the sides. The two narrow sides are also supported on the inside by cross pieces. All the sides are strengthened by supports. When the several parts are put together the bolts, screw-cut at the other end, have only to be inserted through the projecting parts of the longer sides, and made fast by the nuts on the end, to form the whole into a solid box. A cloth spread over the bottom prevents any soap from passing the holes through which the lye drains off.



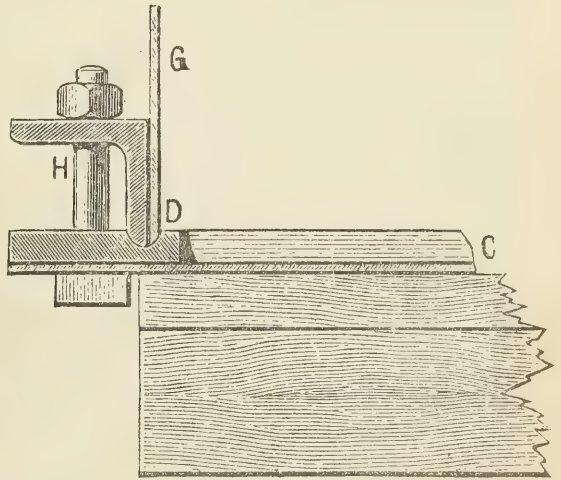
*Frames of iron.*—They are of the same form as the wooden frames, and are best constructed of stout sheet-iron properly strengthened to prevent bulging out. Figs. 41 and 42 show an iron frame constructed of best sheet-iron about 0.17 inch thick and strengthened by flat-irons, angle-irons, and **T** irons. It consists of four side pieces and a bottom and can be taken apart. Its construction is as follows: The sides and bottom are suffi-

Fig. 41.



Iron Soap-Frame.

Fig. 42.



Connection of the Sides with the Bottom of the Frame.

*A*, iron frame; *B*, brass-cock; *C*, bottom plate; *D*, grooves in the flat-irons; *G*, side; *H*, screws.

ciently strengthened by angle-irons and stays of **T** iron. The actual iron bottom is fastened to a second wooden bottom of boards about one inch thick, the whole resting upon strong wooden beams, or upon wheels. The manner of stiffening the frame, especially the number and thickness of the stays of **T** iron, depends on the dimensions, frames with a capacity of 4400 pounds being, for instance, provided with four stays, those holding 8800 pounds with six, and so on.

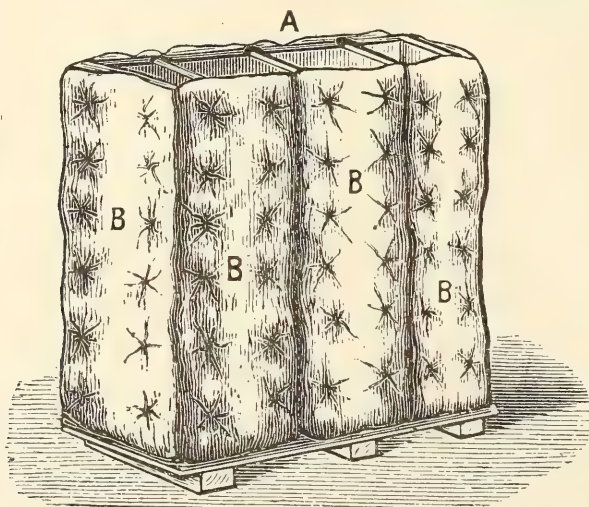
The long screw-bolts on the top of the frame serve to keep the sides at a proper distance from each other, so that the block of soap formed in the frame presents an exactly rectangular shape and perfectly level surfaces.

An absolutely tight and close joint is effected by the half-round and exactly planed edges of the angular irons fitting into grooves also exactly planed in the flat-irons as shown in Fig. 42. The sides are connected with each other and with the bottom by screw-bolts about  $2\frac{3}{4}$  inches long.

The frame is put together in the following simple manner: The workmen place first one of the long sides of it in the groove of one of the long sides of the bottom plate. While one workman supports the side, the other fastens it to the bottom by pushing from below one of the short screw-bolts through each of the two bolt-holes and tightening the screw. One of the workmen then places one of the short sides, which he can easily carry, exactly in the groove of one of the short sides of the bottom, securing it to the bottom in the same manner as above and to the long side already in position, by pushing a screw-bolt through the upper and lower holes. The other short side is then secured in the same manner, and finally the second long side.

Iron being a good conductor of heat, and the soap consequently cooling off quite rapidly, which is injurious to mottled soap, the

Fig. 43.



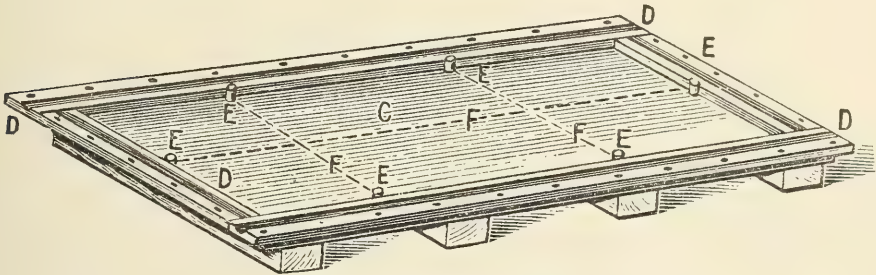
frame is covered with as bad a conductor of heat as possible, which is best effected as shown in Fig. 43 by a mattress of strong jute cloth stuffed with tow. For soaps not requiring mottling, the cover is of course not used, or only if required.

On the bottom of the frame is a further arrangement for draining off and discharging the sub-lye; the latter is effected through the brass cock *B*, Fig. 41.

The bottom of the frame can also be arranged so as to facilitate the execution of the perpendicular cuts in the subsequent cutting up of the soap-block.

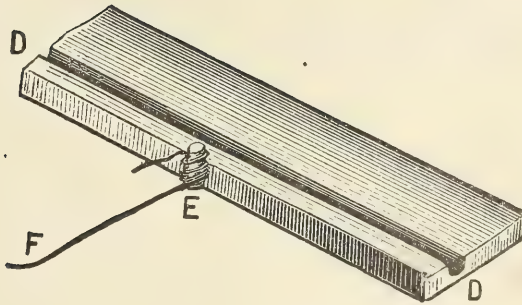
This arrangement is shown in Fig. 44. Small pins, *E*, are set opposite each other in the bottom at a distance of about 0.39 inch from the inner edge of the bottom plate. Previous to filling the frame, cords are fastened to these pins in the manner shown

Fig. 44.



Bottom of Soap-Frame.

Fig. 45.



Section of Bottom Plate.

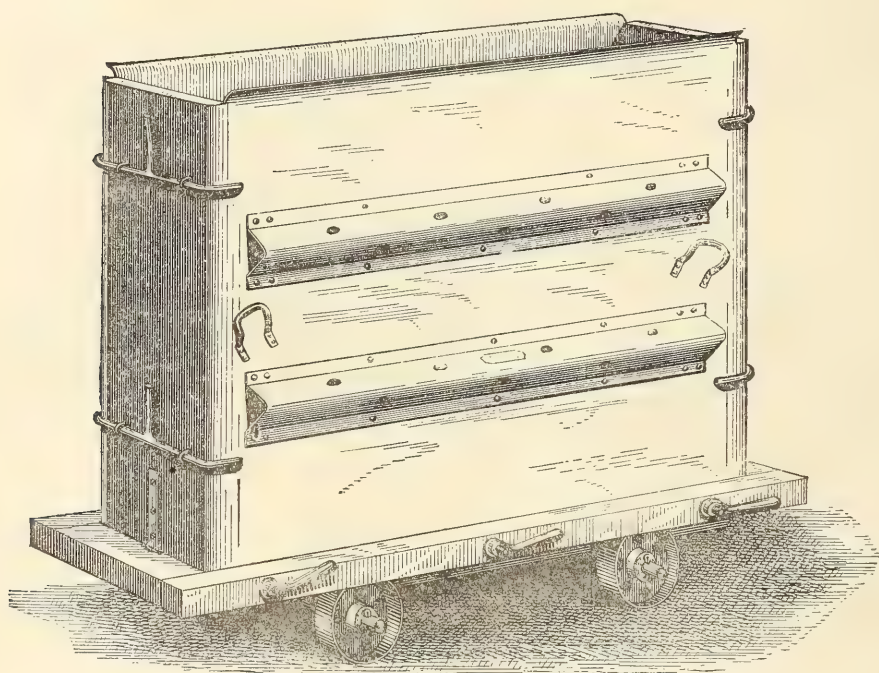
*C*, bottom plate; *D*, grooves; *E*, small pins; *F*, cords.

in Fig. 44. When the soap is to be cut up the wire is drawn under the block by fastening to one end of it the cord previously unwrapped from the pin. This method is very practical and preferable to that in general use of drawing the wire through by means of a hooked iron rod.



Fig. 46 shows Whitaker's patent soap frame, manufactured by Hersey Brothers, of Boston, Massachusetts. It consists of two sides of plate-iron, flanged at their upper edges and strengthened by ribs of corrugated plate-iron, riveted to their outer surfaces and running in the direction of their length. These corrugations prevent the bending or twisting of the side plates, and the soap

Fig. 46.



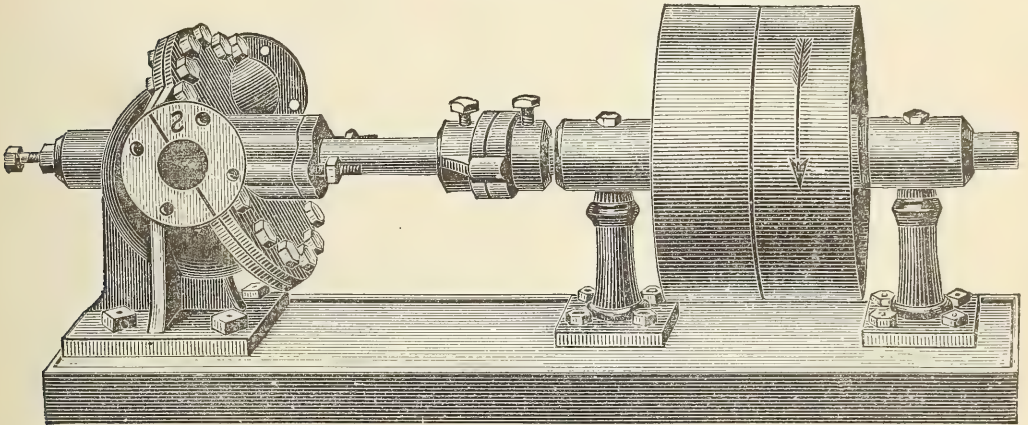
cools into the exact rectangular shape of the frame. The trouble and expense of the ordinary stays and supports are here avoided, as the frame is self-sustaining. The sides are connected by ends of two-inch plank, secured by clamps. The frame is very light, easily managed, and can be adjusted and taken apart by one workman almost momentarily. The soap cools very rapidly; ordinary soap cooling sufficiently to strip in twenty-four hours in cold, and in forty-eight in warm weather.

The frames for toilet-soaps differ from those for ordinary soaps only in size.

*Soap-pumps.*—Hersey's patent rotary soap-pump, manufactured by Hersey Brothers, of Boston, used in many large factories in the United States, is shown in Fig. 47. The pump may be

set up in any convenient position adjacent to the kettle, and not over 10 feet above the bottom of the same, and connected to it by means of a  $2\frac{1}{2}$ -inch iron pipe, tapped through the iron plate at a distance of about 2 feet above the worm or coil. Each kettle is thus connected with the pump by the iron pipes, which have valves placed upon them on the outside (of the kettle), so that the contents of any one of the kettles may be readily pumped off and framed without disturbing the others. The pipe inside of the kettle has a suitable swing-joint so arranged that it can be raised and lowered at pleasure. Fig. 47 represents the pump complete. When the pump is rotated in the direction of the

Fig. 47.



arrow, the outlet marked *S* is the suction; when rotated in the opposite direction, the opposite outlet becomes the suction. This is an important feature, as it enables the discharge-pipes to be emptied of their contents in stopping, by giving a few revolutions by hand in the opposite direction. Fig. 48 is a view of the interior of the pump when the cover is taken off. When turned in the direction of the arrow the blade *F* sweeps around, drawing the fluid in at *I* and forcing it out at *H*, the contents of the pump being twice emptied during each revolution. The blade *F* swings on a pivot; the end of *F*, when reaching the point *B*, at the lowest point, gaining the position there shown, and gradually returning to its former position on completing the revolution. The fluid is prevented from passing from one side to the other by the contact of the cone with the cover. The set-screw shown in the

illustration bears against a step at the end of the cone and keeps the cone forced against the cover, and is screwed up to compensate for any wear that takes place. Fig. 49 shows the cone and blade and forms the entire working part of the pump. No valve

Fig. 48.

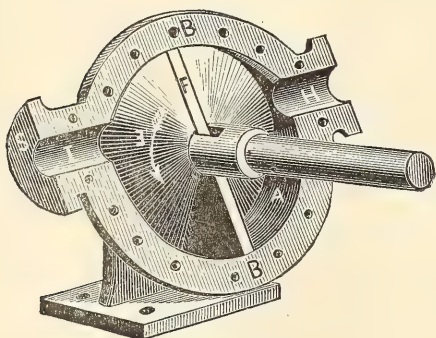
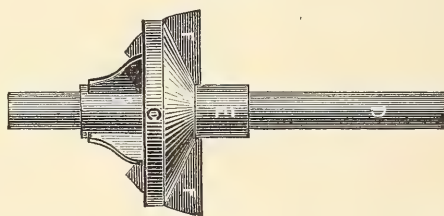


Fig. 49.

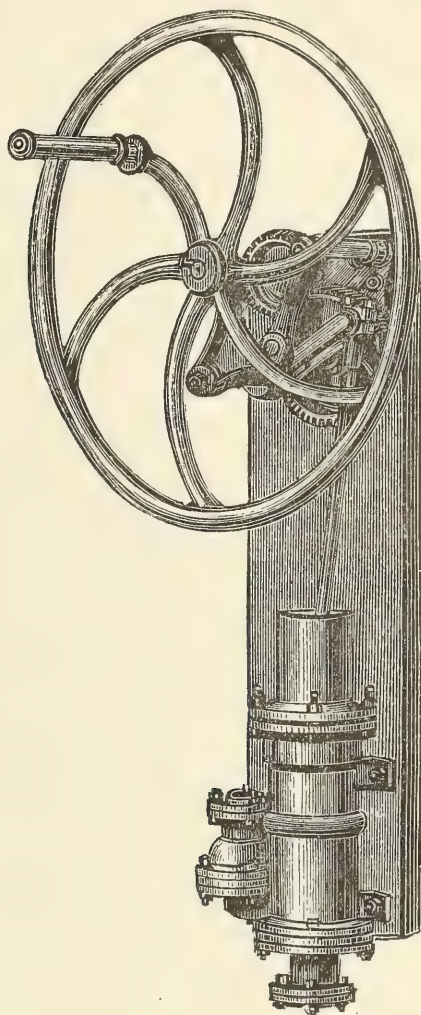


being used, there is no chance of any derangement in the operation of the pump. The soap can be forced any distance or height. It can be pumped from one kettle into another, which is a special advantage when it is necessary to transfer the "nigre," either in the state of a soft curd or in the unseparated state, into another kettle to make room for a fresh boiling with clean stock, and thereby keep up a uniform quality of first-class soap. By lowering the pipe attached to the swing-joint inside of the kettle, the lye of the "strengthening change" can be pumped from the very lowest point in the bottom of the kettle, while still hot, into another kettle in which stock is being saponified, thereby economizing steam. In certain cases where it is undesirable to pump the lye over the curb into the kettle, because of the froth which it may occasion, another plan can be adopted, which admirably brings into play the whole system of pump, valves, and swing-joints of the two kettles, from the bottom of one of which it is required to pump the hot lye and force it through the iron piping down to the bottom of the other. Lye of any kind, whether spent lye or strengthening change lye, strong caustic lye, either hot or cold, grease or thick oil, can be easily and quickly pumped by this pump. The pump is 10 inches in diameter and  $2\frac{1}{2}$  inches in outlet, revolutions 120 per minute, capacity 6000 gallons per hour.



The pump shown in Fig. 50 is well adapted for smaller factories. It is fastened to a board on the wall near the kettles by means of screws or iron bolts. It is provided with a fly-wheel

Fig. 50.

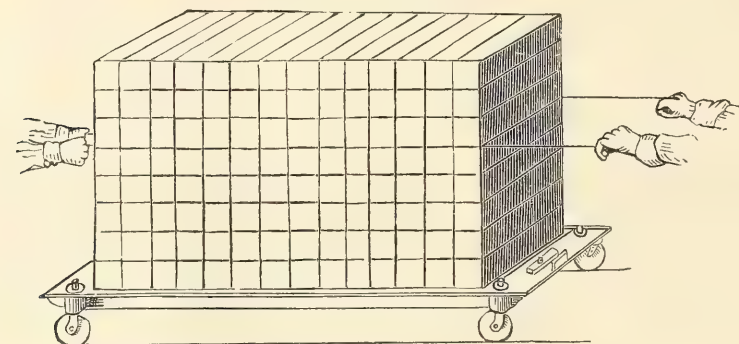


and gearing and arranged so that it can be worked by hand or power. It has a piston with a diameter of about 4 inches and a stroke of about 10 inches. A suction-pipe is screwed to the lower end of the pump and can be placed in any of the kettles. The pump pumps both soap and lye.

*Soap-cutting machines.*—When the soap sets firmly, the frames, according to their construction, are either lifted off, or taken apart and removed, so as to leave the solid block of soap resting upon the bottom. The block is then divisioned off by means of a

scribe, an instrument of hard wood smoothly planed and provided with slender iron teeth set at the thickness desired for the bar of soap. The workman then taking brass or steel wire fastened to a wooden handle draws it through the block, Fig. 51, which is

Fig. 51.



thus divided into slabs. When the whole is thus divided, the slabs are removed and reduced in their turn to bars and lumps of smaller dimensions. The usual size of the bars was formerly 12 to 14 inches long by 3 inches every other way, but at present they are mostly cut into one pound and one-half pound blocks, a form which also prevails to a very large extent in England and on the Continent.

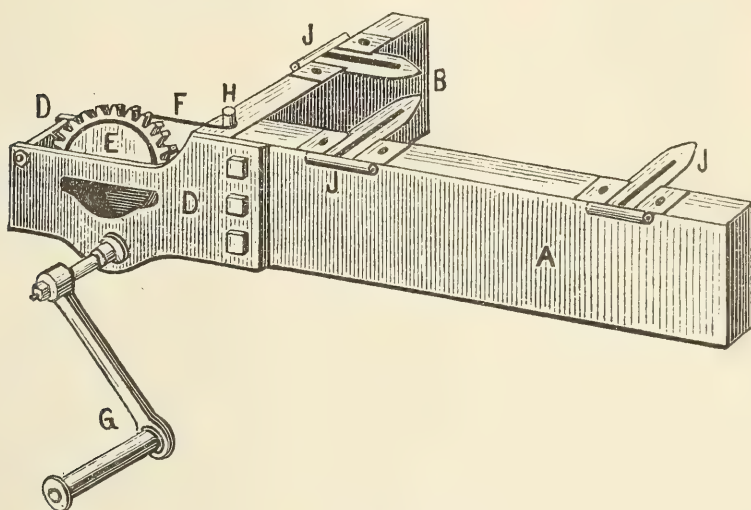
In modern times many machines for cutting the block of soap into slabs and bars have been constructed, a few of which shall be described.

Figs. 52 and 53 show Krull's soap-cutter. This apparatus, weighing only about  $17\frac{1}{2}$  pounds, is convenient to handle, and by a simple contrivance can be directly placed and secured to a block of soap of any size without injuring it. Further, it is so constructed as to take up but little room, being therefore especially suitable for small factories. The workman can execute the cut through a block of soap of any size with great ease and in a short time, and besides the cut is exactly straight, thus preventing crooked and irregular cuts and the consequent waste unavoidable in cutting by hand. The apparatus allows of making horizontal and vertical cuts.

The construction is very simple. The apparatus consists of two boards, *A* and *B*, joined at a right angle, which carry the

actual cutting arrangement. This consists of the cutting-wire *F*, which wraps around the cogged drum *E*, placed between the two iron escutcheons *D* and set in motion by the crank *G*. One end of the cutting-wire *F* is secured to the drum, and the other end

Fig. 52.



Krull's Soap-Cutter.

*A*, *B*, boards; *D*, iron escutcheons; *E*, cogged drum; *F*, cutting wire; *G*, crank; *H*, pin; *J*, slide for fastening the apparatus.

twisted into an eye is hooked over the pin *H*. The entire apparatus is fastened to the block of soap by means of the three slides *J*.

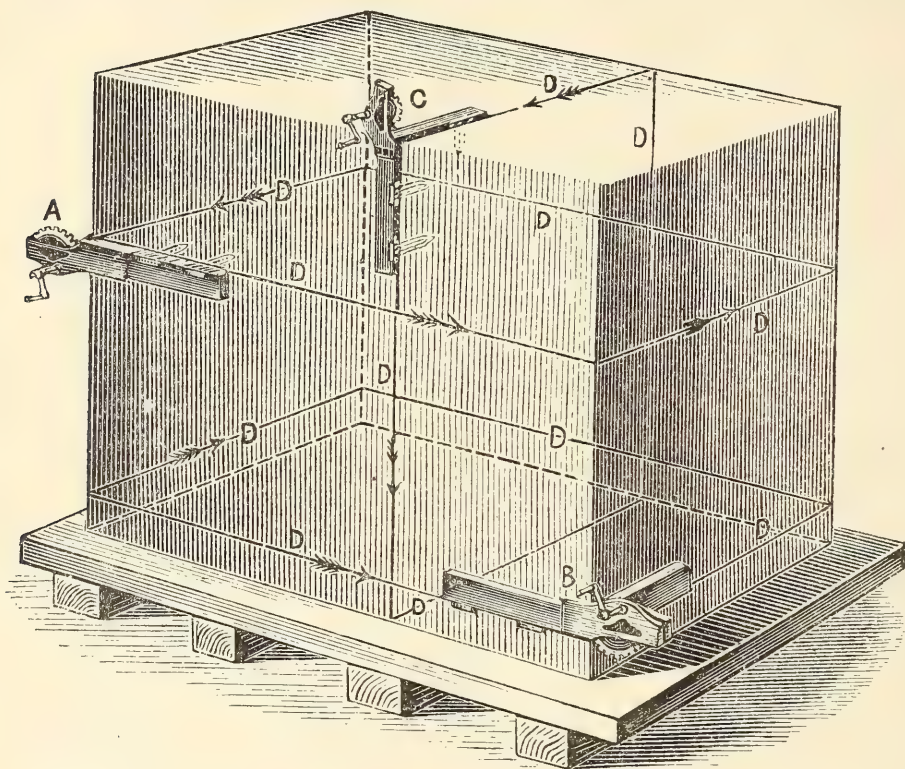
The apparatus is used in the following manner: After divisioning off the block by means of a scribe in the same manner as for hand-cutting, the apparatus is secured to the block by means of the slides *J*, the cutting wire placed around the block and the free end twisted into an eye hooked over the pin *H*, thus forming a loop, which by turning the crank becomes smaller and smaller until the wire has passed entirely through the block. Fig. 53 shows the arrangement for horizontal cuts, *A*, vertical cuts, *C*, and for cuts, *B*, near the bottom of the frame.

Fig. 54 shows the iron soap-cutting frames, with adjustable wires, manufactured by Wm. H. Dopp & Son, of Buffalo, N. Y., and the manner of using them. Make the frame as indicated by the cut of 3 × 4 scantling and firmly bolt on the iron frames. Make the bed-plates of one-inch planed stuff, and fasten to the



left hand side of two of them a wooden ledge. Place one of these in front of each iron frame in space marked with arrow.

Fig. 53.



Krull's Soap-Cutter; manner of executing the cuts.

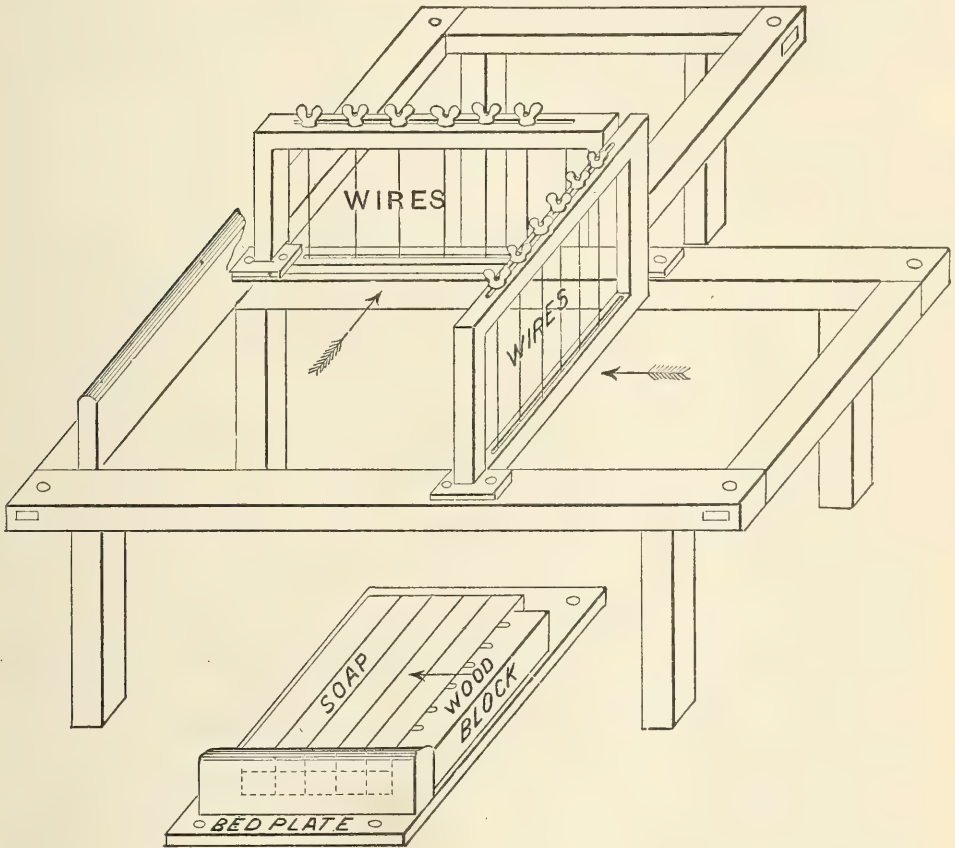
A, horizontal cuts; B, cuts near the bottom of the frame; C, vertical cuts.

Fasten them with wood screws so that they do not shift, but can easily be taken off by unscrewing the screws. This is necessary in putting in extra wires or taking out superfluous ones. On the space behind the frame, on left side of cut, put a bed-plate without ledge on side. The wires can be set any desired distance apart by means of the thumb-nuts.

The soap is cut in the following manner: Having removed the sides and ends of your soap frame from the block of soap, mark off on both sides and ends of the block the thickness of the slabs desired. Now let two men take each one end of a long steel wire attached at each end to a wooden handle. Place the wire against the first line marked on end of block of soap, and pull the wire horizontally through the soap to the other end, one man walking

on each side of the block. In the same manner return and in this way cut the block in slabs. Now place the wire across the top of the block 16 to 18 inches from one end, and pull it vertically downward till it reaches the base board; then draw it

Fig. 54.



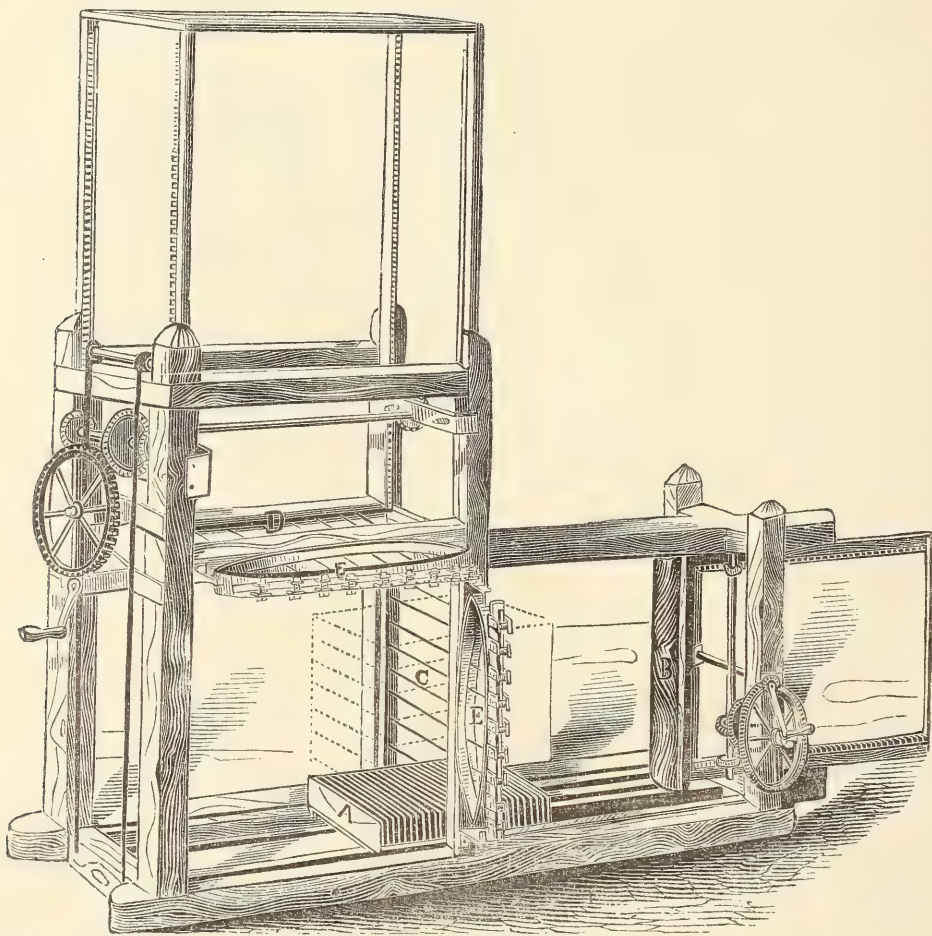
horizontally along the base board for 16 to 18 inches, and then vertically upward. In this manner cut the long slabs into short slabs of such length that they will pass through the iron frames and can be easily handled.

Place as many slabs as a man can conveniently push on the bed-plate to the extreme right, and behind the soap a wooden block having as many notches cut in it as are wires in the frame. These notches must correspond with the wires in position, and must be deep enough to allow the soap to be pushed clear of the frame. Take hold of the block and push the soap through the first frame. Then with a block corresponding to the wires in the second frame, push the soap already cut into strips through this

frame and it is cut into cakes. It is really astonishing how much soap a man can cut with this simple device in a day. The whole machine complete should not cost more than \$15 to \$20.

A very rapid method of dividing the blocks into bars is by the use of two pieces of machinery, Fig. 55 and Fig. 56, the first being called the *slabbing and barring* machine, and the second the *caking* machine. Fig. 55 consists of a carriage *A*, which is

Fig. 55.



Slabbing and Barring Machine.

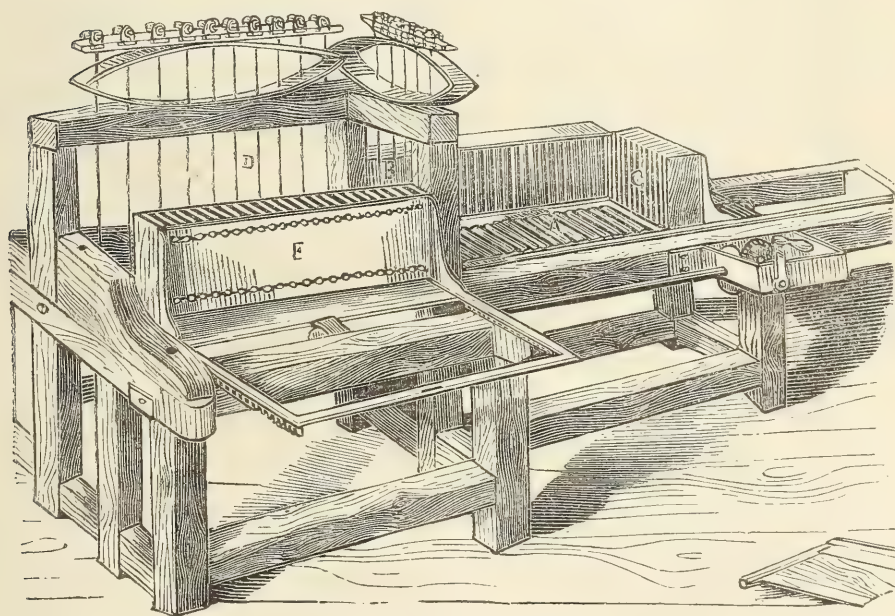
so grooved at the top as to allow the wires to pass entirely through the block of soap. This carriage is then moved back to the driver *B*, and on it is placed a whole block of soap as it comes from the frame. This is done by a peculiar truck, as shown in the illustration, constructed expressly for the purpose. The block of soap having been first cut loose from the bottom of the frame,



this truck is run to the side of it, and, by means of rack and pinion worked with a lever, the block of soap is slipped on the truck, brought to the machine, and, by the same power, placed thereupon.

The range of wires *C* is regulated by corresponding gauges in the upright posts, which allow it to be set to cut slabs of any desired thickness. The block of soap is forced up to the wires by the driver *B*, propelled by means of racks and pinions and a winch. It will be seen that in this way the block will be converted into slabs. There is a similar horizontal arrangement of cutting-wires *D*, and confined to a vertical motion by the posts of the frame. These wires are also arranged as above, so that any desired bars may be cut. They are caused to descend by the action of the rack and pinions and winch as above; and with this part of the machine, the slabs are converted into bars without handling them. They, consequently, are much neater and smoother than they could be if otherwise cut.

Fig. 56.



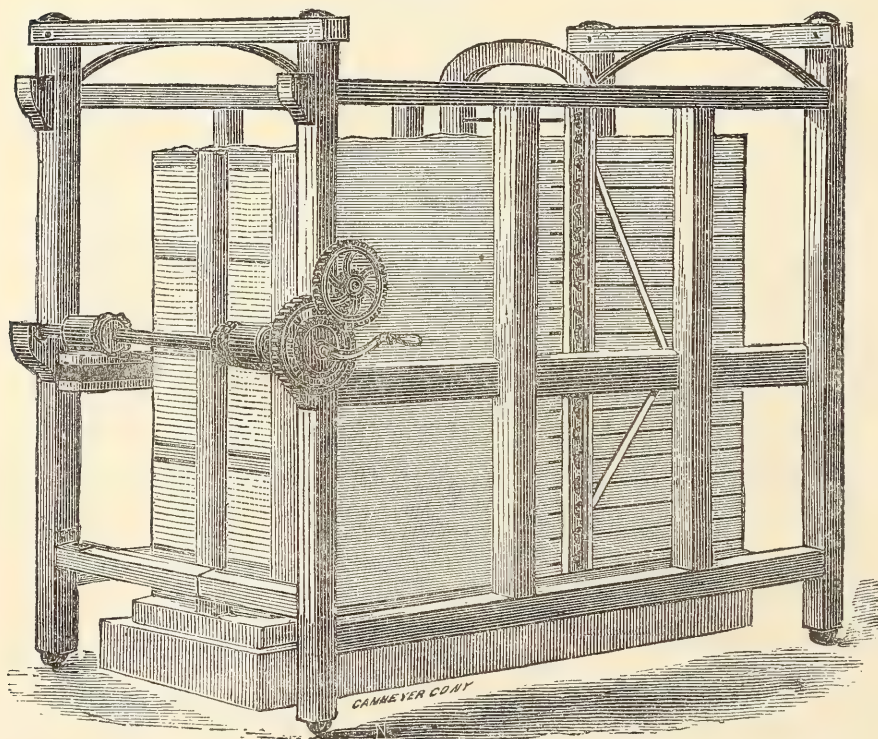
Caking Machine.

The wires being fastened at one end to a spring *E E*, will easily yield and form the required loops at the beginning of the operation; and then both ends become fixed so that the loops cannot

get any larger if the soap be very hard ; in which case the long loop is more apt to warp and cut uneven. The steady motion of this machine permits the use of much smaller wire than will do for hand-cutting, and, consequently, the work is much smoother. This apparatus cuts the blocks of soap into bars as long as its width. To make pound lumps, or small cakes and tablets, the slabs must be transferred to the *caking machine*, Fig. 56.

The slabs are placed, in as great number as can be got on, upon a range of rollers *A*, and forced through the range of wires *B* by the driver *C*, which is propelled by racks and pinions and a crank. The soap having been forced through lengthwise, and the crank being shifted, it is then forced through the range of wires *D* by the driver *E*. Both the drivers are connected with the same crank, and by displacing the latter from the one it gears itself into the other. The wires are arranged in the same manner as in the slabbing machine. They may be readily shifted so as to cut any desired shape or size.

Fig. 57.



This mode of cutting gives great smoothness and uniformity of weight and size to the bars and lumps, saves handling, scratch-

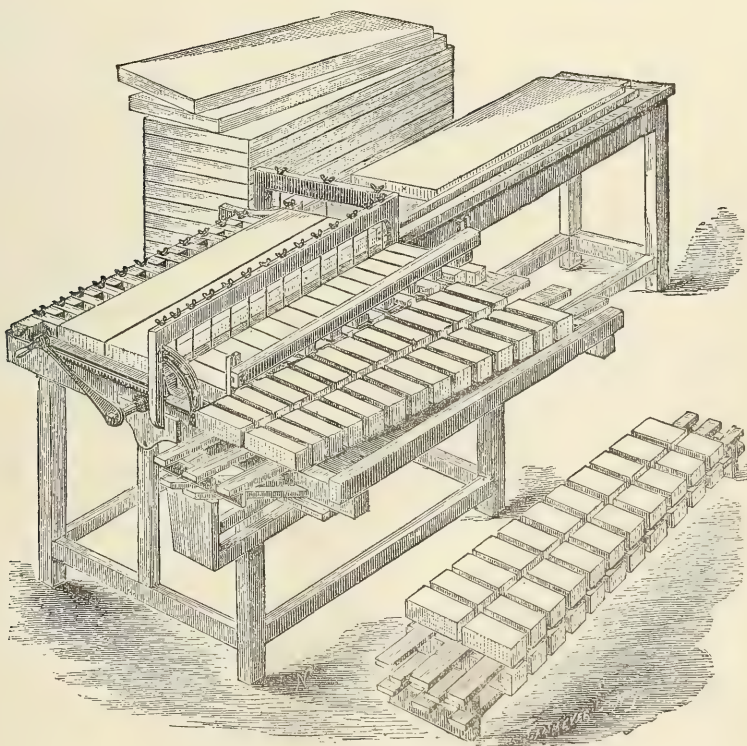


ing, and bending, and effects a larger gain over the usual method in time, labor, and expense.

Fig. 57 shows a slabbing machine, and Fig. 58 a cutting machine, manufactured by A. W. Houchin, of Brooklyn, New York. The slabbing machine has a movable head and the wires are drawn through the soap by compound gearing with a steady pull, all the while in operation, thus leaving the slabs perfectly level and smooth, each and every one of the same thickness, with a fine lustre, after the wires have passed through the soap.

The cut (Fig. 57) shows the soap in the machine and the wires partly drawn through. When cut through, the hinge on the back end is opened, the machine drawn from the soap, then the head is run back, shaft placed in the boxes above, hinge folded in front and drawn to another frame; then the hinges are closed, the shaft

Fig. 58.



placed in the former boxes, and the head-block placed between the shaft and the front of the soap, when the machine is ready to cut another frame. The whole process for getting ready and cutting a frame of soap can be done in less than five minutes.

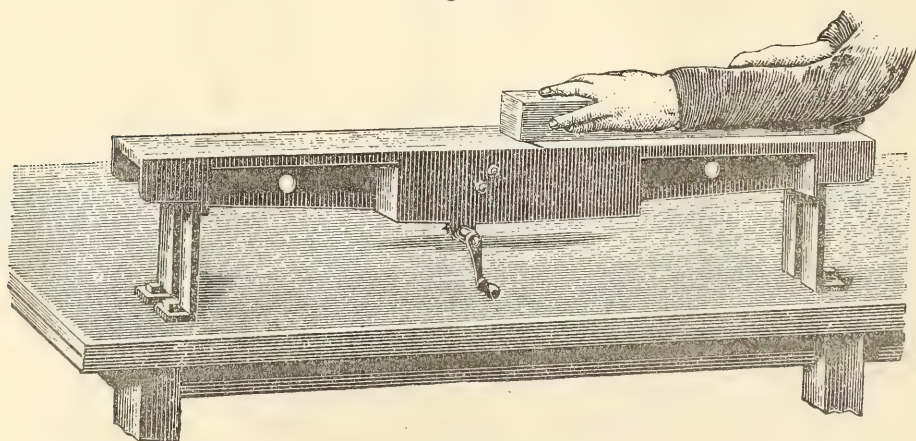


Fig. 58 represents the machine for cutting, trimming, racking, and spreading the soap ready for drying, leaving all the bars or pieces of an equal size and weight, smooth and clean, without any finger-marks.

First the slab is placed on the table, then drawn through the wires endwise, trimmed, and then passed through wires that cross-cut it into bars, and then left on the racks, which, by being drawn endwise, spreads the soap in a uniform manner, all the bars at equal distances apart, ready for setting away to dry and box.

*Soap-planes.*—To give the soap a better appearance it has to be equalized and planed. This is frequently done by a plane laid upside down and firmly secured to a table. Fig. 59 shows such

Fig. 59.



Soap-Plane.

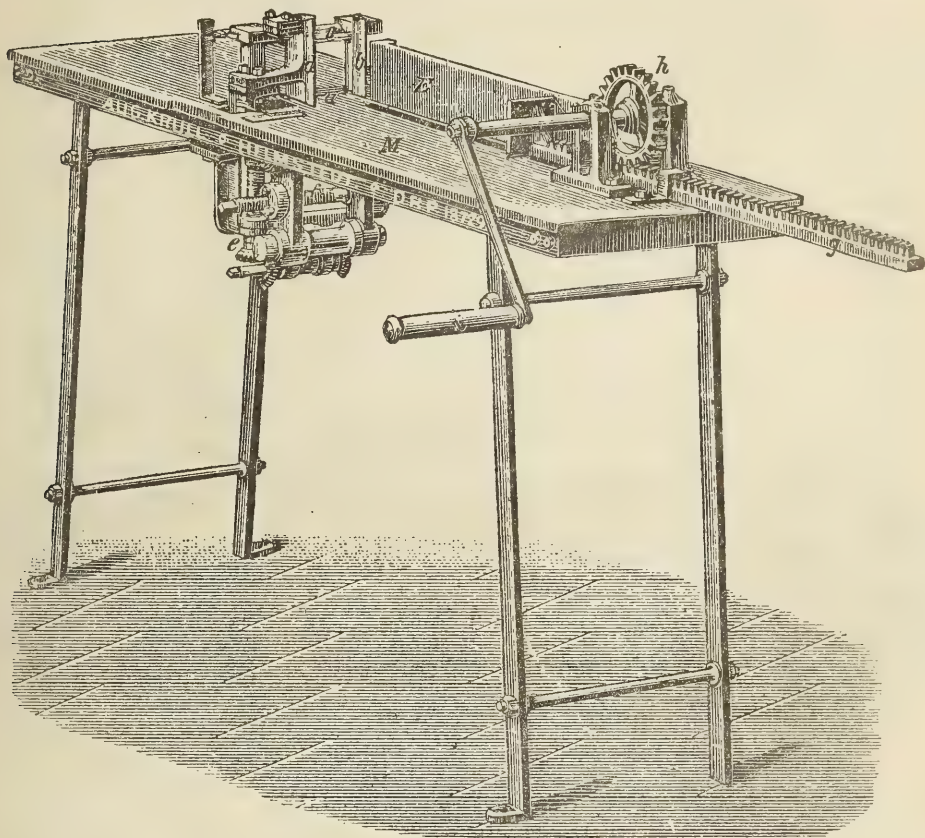
an arrangement, entirely constructed of steel and iron. The knife can be accurately set by means of a spindle.

It is clear that with this instrument only one side of the bar can be equalized and planed at one time, and though, with some skill, the bars acquire a neat appearance, their dimensions are not exactly alike.

To be enabled rapidly to plane large quantities of bars which shall show exactly the same dimensions, such planing-machines as are shown in Figs. 60 and 61 have been constructed. They are provided with four knives, so that the bar, after passing through the machine, is planed and polished on all four sides. The construction of the knives allows of the planing and polish-

ing of all kinds of soap and even of bars which have become warped and crooked in drying. The planing mechanism of the two machines is exactly the same, they differing only in the construction of the mechanism which propels the bar of soap forward. In the machine shown in Fig. 60 this is effected by means

Fig. 60.



Soap-Planing Machine.

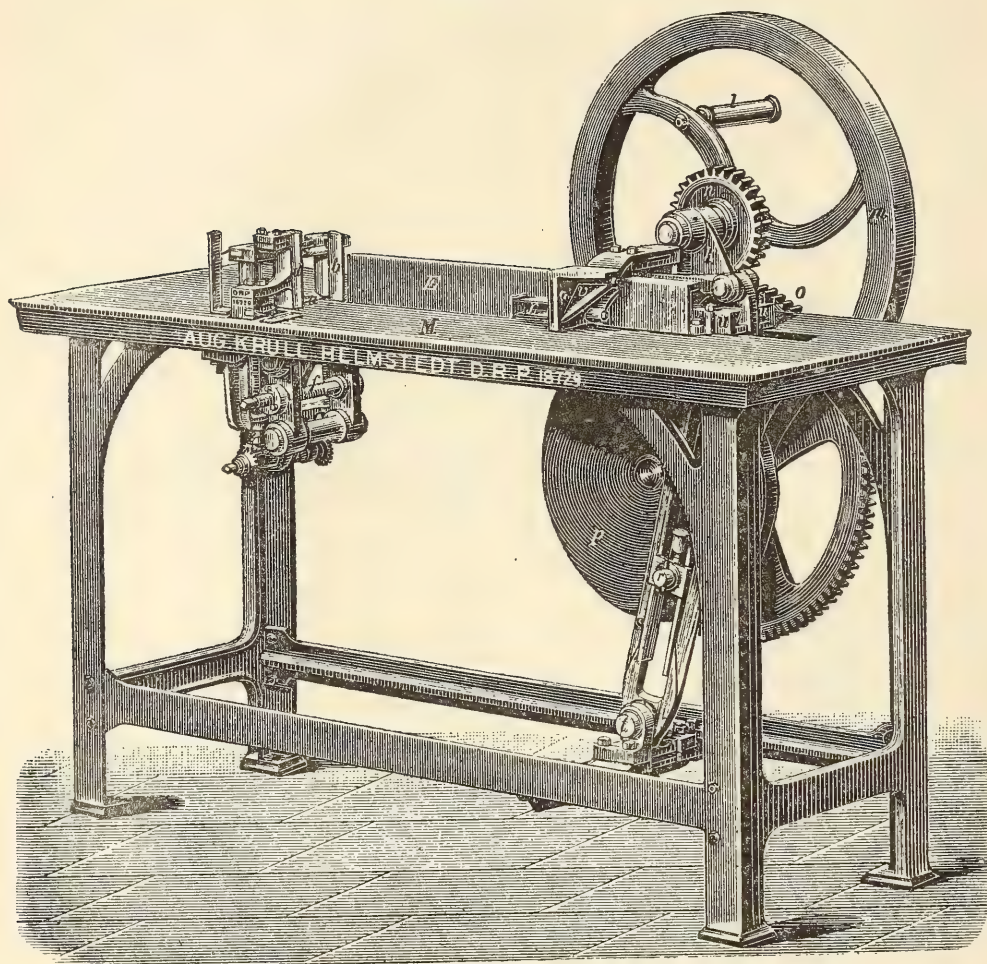
of rack and pinion and a crank, and in Fig. 61 by means of an oscillating crank-loop with spur-gearing. This arrangement allows of the machine being fitted for steam-power.

The above machines are manufactured by August Krull, of Helmstedt, Germany.

*Crutching machines.*—Stephen Strunz's soap-crutching machine (Figs. 62 and 63) is simple in construction and perfect in its action. It is manufactured by Messrs. Hersey Bros., of Boston. It crutches the soap completely within three minutes and gives it great smoothness and transparency. Size, 1200 pounds.



Fig. 61.

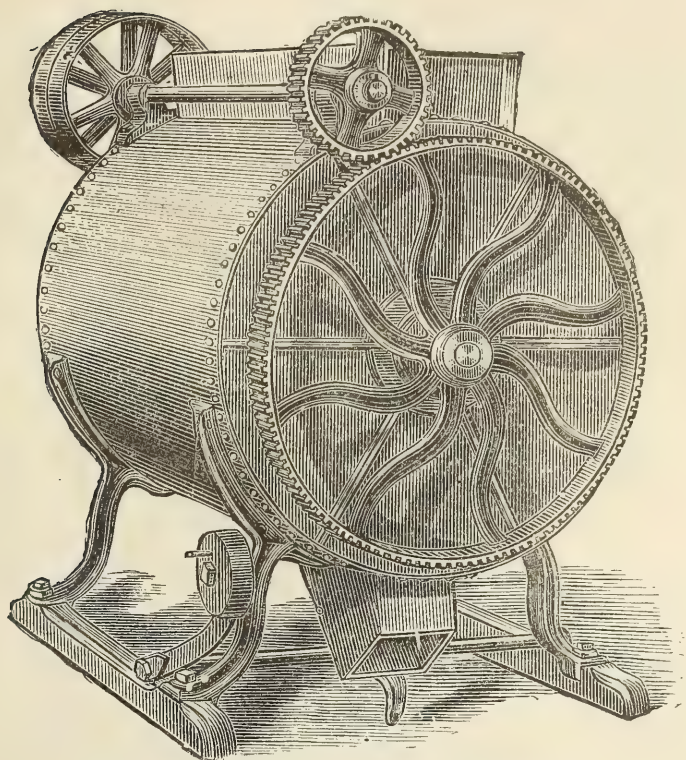


Soap-Planing Machine.

The speed of the main shaft, with the working paddles on, should be 45 to 50 revolutions per minute, and turn so as to work the soap to the valve and pump it out. When the machine is charged, the soap should cover the paddles two inches before the machine is started. When running the soap into the frame, the machine should be stopped until the soap commences to run slowly, otherwise it will force it out too rapidly. Very little power is necessary for this machine. To clean it put in four or five bucketfuls of boiling salt water of about  $22^{\circ}$  B. and run the machine three to four minutes, which should be done while the soap remaining in the machine is warm. The machine must always be cleaned after using.

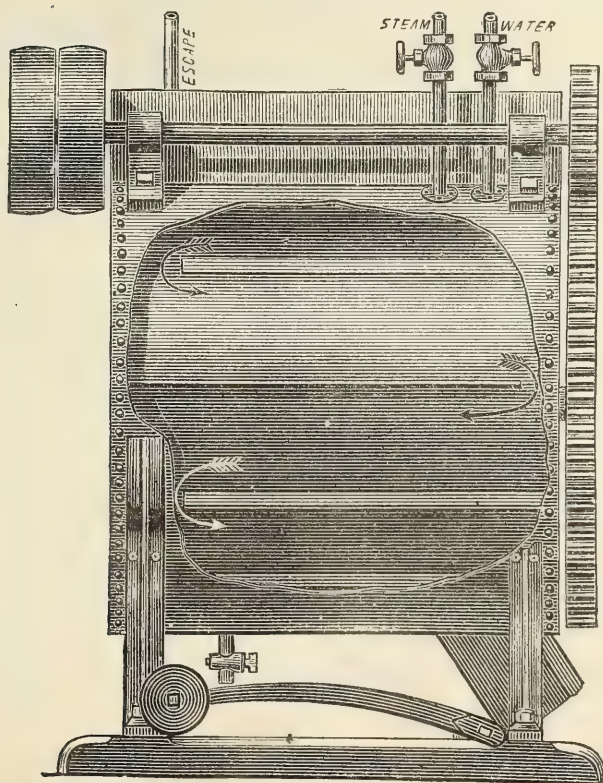


Fig. 62.



Strunz's Crutching Machine (outside view).

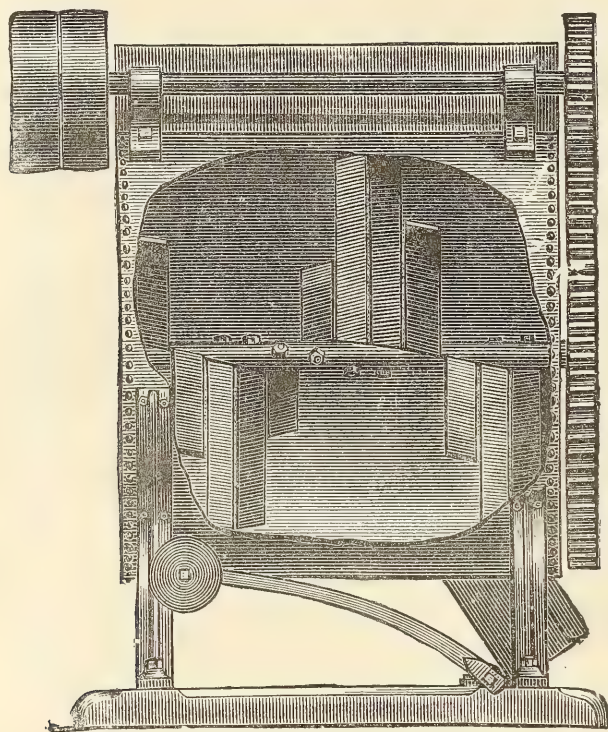
Fig. 63.



Strunz's Crutching Machine (working part of machine).

*The jacket crutching machine.*—The jacket on this machine (Fig. 64) is a circulating one, and is said to have no equal in its rapid heating or cooling power. There is no dead point, as in other jackets, where the warm water remains for a long time outside the current. The drawing shows two pipes on one side; one is to be connected with steam and the other with water. The

Fig. 64.



Strunz's Jacket Crutching Machine.

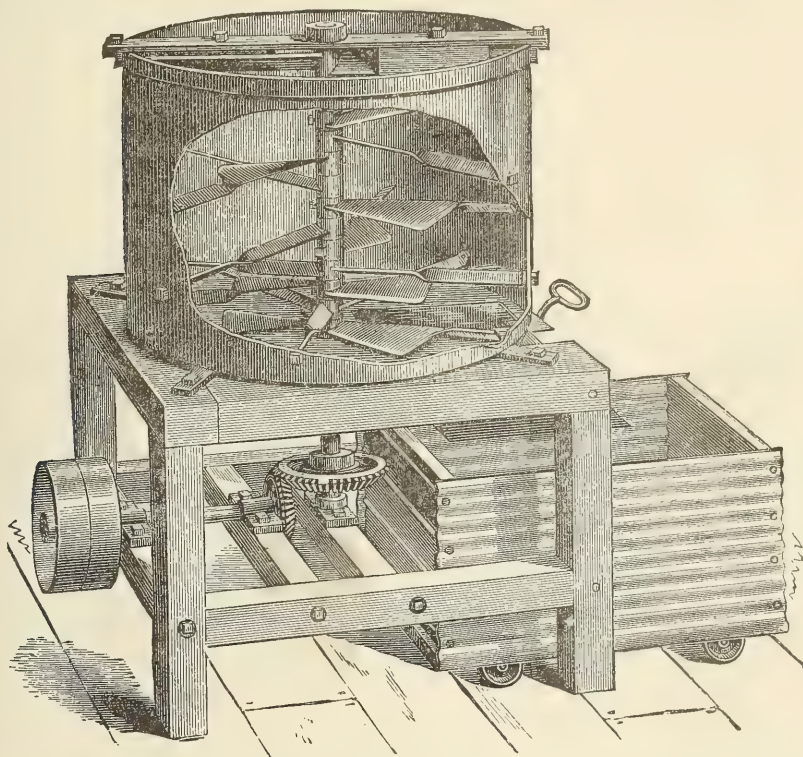
escape should be left always free and no cock or valve should be on the escape-pipe. The little cock on the bottom is to let the water out of the jacket to prevent it from freezing, or else the jacket would burst. The steam should never be let on unless the jacket is free from water, otherwise it may strain the machine.

Fig. 65 represents the improved soap crutching machine manufactured by Alfred W. Houchin, of Brooklyn, N. Y. The vertical wrought-iron shaft, which is rotated by the gearing shown beneath, carries a number of cast-iron wings, smaller extensions of which project to the inner periphery of the containing vessel. Within the latter are also a number of fixed bars, which are



rigidly secured, and pass through sockets at the inner ends of which the shaft freely turns. The wings are constructed in a spiral form, and work as a double acting screw, raising and mixing the heaviest material from bottom to top. No air is crutched into

Fig. 65.



the soap, as the stirring is all within the substance, so that it cannot get a spongy appearance or become filled with air-holes and blisters. The product looks like the best hand-crutched soap, and is perfectly smooth and firm.

In operation the soap is let into the top of the tank, and run by steam power from four to ten minutes; the sliding door in the bottom being then opened the material is conducted to the frame, placed as shown in the illustration, to receive it.

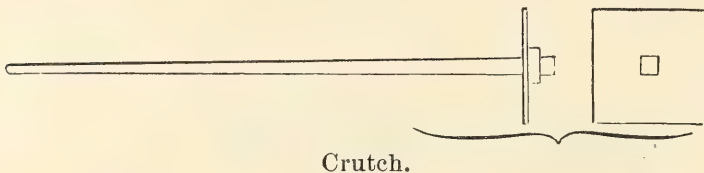
The crutching machines, above described, are very useful for mixing the colors and perfumes for toilet soaps.

Almost all soaps are now stamped and many wrapped; for stamping there are numerous presses in use, the most important of which will be described in the chapter on toilet soaps.



*Minor implements.*—The minor implements of the soap factory are, a crutch, Fig. 66, composed of a long wooden handle fixed

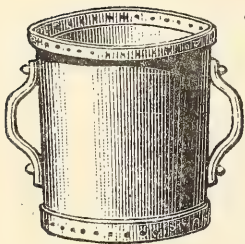
Fig. 66.



Crutch.

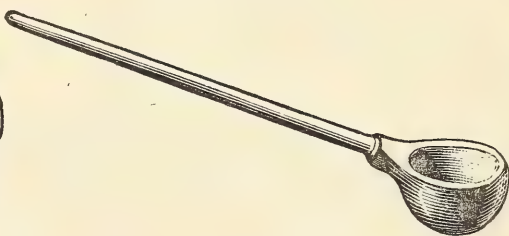
to a board, and used for stirring the soap paste; copper dippers with handles (Figs. 67 and 68) for dipping the hot paste from

Fig. 69.



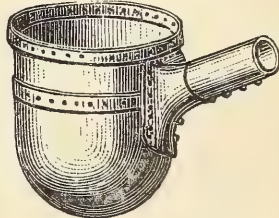
Bucket.

Fig. 67.



Dipper.

Fig. 68.



Dipper.

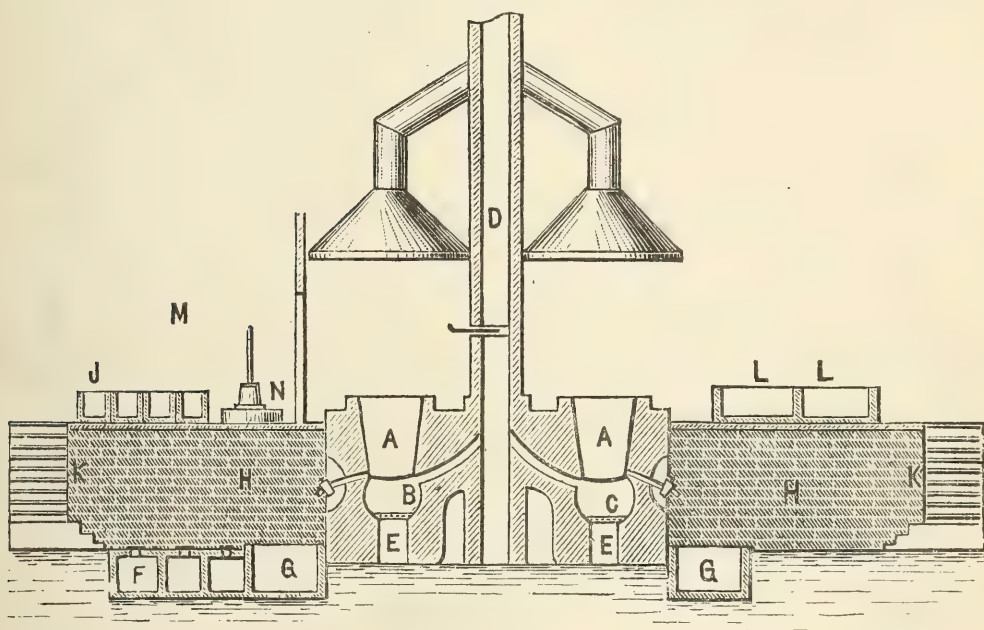
the kettles, and copper buckets (Fig. 69) for conveying it to the frames.

*General plans of soap factories.* 1. *With the use of an open fire* (Fig. 70).—The building has a rectangular form, the dimensions of which vary according to the importance of the manufacture. It is divided into three compartments, the middle one being occupied by the kettles *A* and the frames; the room on the left contains the lye vessels *J*, and that on the right is employed as a store-room. The stairs *K* lead to the basement, a part of which is occupied by the furnaces, the oil reservoirs *F*, and a cistern of masonry *G* to receive the waste lye drawn from the kettles.

The kettles *A*, which serve for the saponification of the fats and oils, are placed on a parallel line. Over each kettle is a hood to conduct the vapors into the chimney *D*. Below these kettles are passages in which are placed the furnaces and masonry vats *G* to receive the waste lyes which collect at the bottom of the kettle below the soap. The fireplace *B* is the space which separates the grate *C* from the bottom of the kettle. The space varies

from 13 to 20 inches, according to the capacity of the kettles. The grate *C* is composed of cast-iron bars placed at the distance of about one-third of an inch apart. These bars are generally one inch in thickness, so that the grate presents a surface of

Fig. 70.



General Plan of a Soap Factory Working with an Open Fire.

*A*, soap-kettle ; *B*, furnace ; *C*, grate ; *D*, principal chimney ; *E*, ash-pit ; *F*, oil reservoir ; *G*, masonry vats for waste lyes ; *H*, cellar ; *J*, lixiviating vessels ; *K*, stairs ; *L*, frames ; *M*, store-rooms for crude soda ; *N*, pulverizing trough,

draught equal to one-fourth of its total surface. Experience has shown these proportions to be the best to assure a complete combustion of the fuel.

Into the chimney *D* all the products of combustion are discharged. The higher the chimney the better the draft. Its inside diameter must always be proportioned to the total opening of the flues of the furnace. To hasten or slacken the combustion in the furnaces the chimney is provided with a register.

The object of the ash-pit *E* is to convey air between the bars of the grate and to serve as a receptacle for the ashes. Its dimensions vary, but it is generally as wide as the grate.

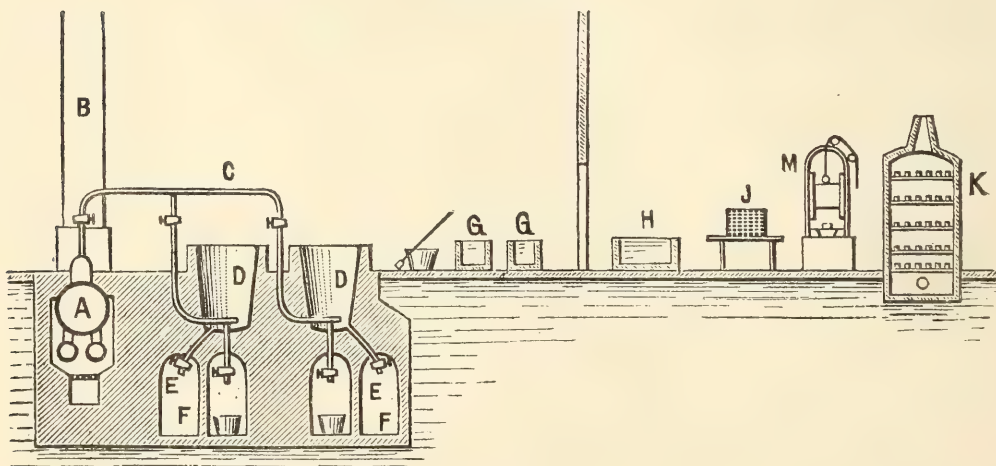
A pump is placed in each of the cisterns *G* to raise the waste

lye they contain into a large masonry or sheet-iron vat placed on the first floor.

The store-room *M* contains the raw materials for the preparation of lyes. The materials are pulverized and mixed with lime in the trough *N*.

2. *With the use of steam.*—The application of steam to the fabrication of soaps has become nearly general. This system presents many advantages over heating by an open fire. Fig. 71 shows a general plan of a factory in which all the kettles are heated by steam. The steam-boiler *A* is placed over an ordinary fireplace with cast-iron grate; the products of combustion escape through the chimney *B*. From the dome of the boiler the steam is discharged through the pipe *C* and conducted to the kettles *D*, which have the ordinary shape, only at the bottom there is a horizontal worm in which steam continually circulates during the boiling of the soap. Each worm is provided with a waste-pipe *L*, which traverses the bottom of the kettle to discharge the water of condensation.

Fig. 71.



General Plan of a Soap-Factory, with the Use of Steam.

*A*, steam-boiler; *B*, chimney; *C*, steam-pipe; *D*, soap-kettle; *E*, discharge-pipe; *F*, masonry cistern; *G*, lye reservoir; *H*, frames; *J*, cutting table; *K*, drying-room; *L*, discharge-pipe for water of condensation; *M*, soap-press.

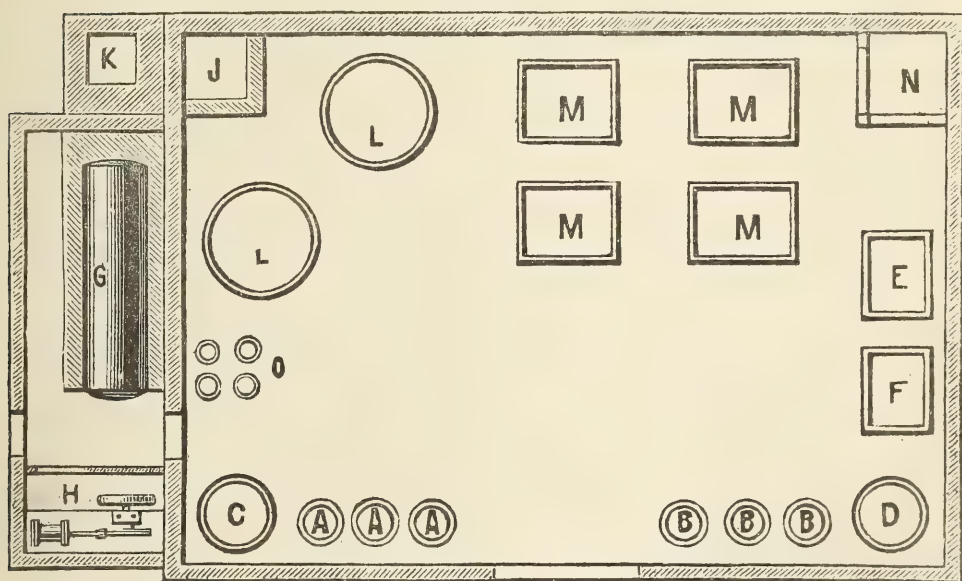
The above-mentioned dome of the boiler is necessary to prevent the boiling water from entering the pipe and thence passing into the coil.



The pipes *E* conduct the waste lye into the masonry cisterns *F*. To render the kettles more solid and prevent the loss of heat, the foundation of the kettles is made of brick and cement. The other arrangements are made clear by the letters in the illustration. The lye to be used is in the reservoir *G*; the finished soap is brought into the frames *H*, and after sufficient cooling upon the cutting table *J*, where it is divided into bars and cakes which are dried in the drying chamber *K*. The bars or cakes are finally stamped in the press *M*.

3. *Ground-plan of a soap-factory, with the use of superheated steam* (Fig. 72).—The factory is located in a rectangular building, the basement of which is divided into two compartments, the

Fig. 72.



Ground-Plan of a Soap-Factory, with the Use of Superheated Steam.

*A*, Lixiviating vats for potash ; *B*, for soda ; *C*, evaporating boilers for potash lye ;  
*D*, for soda lye ; *E*, reservoir for potash lye ; *F*, for soda lye ; *G*, steam-engine ;  
*J*, steam superheater ; *K*, chimney ; *L*, soap-kettle ; *M*, frames ; *N*, hoist ;  
*O*, barrels for soft soap.

smaller one of which contains the steam-engine  $H$  and the steam-boiler  $G$ , and the larger one three vats,  $A$ , for potash lye and three vats,  $B$ , for soda lye, with the requisite evaporating boilers,  $C$ , for potash lye, and,  $D$ , for soda lye. The finished potash lyes are brought into the reservoir  $E$  and the soda lyes into the reser-

voir *F*. Two soap-kettles stand near the superheater *J*. From these kettles the finished soaps can be readily brought either into the frames *M* or the barrels *O*. By means of the hoist *N* the soap blocks are conveyed to the first floor, which contains the cutting machines and drying rooms.

The materials for the preparation of the lyes, the fats and oils as well as the finished soft soaps, are stored in the cellar.

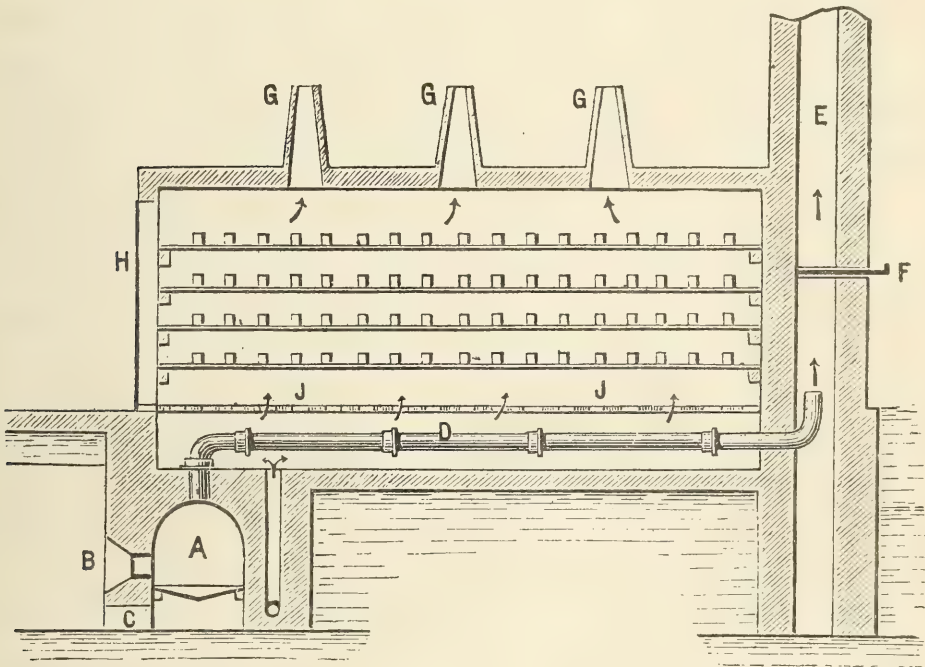
*Drying-rooms.*—All soaps do not require drying, but as many do, a drying-room is very necessary—one with warm air or steam heat, or one well ventilated by air. The latter does not require any heating apparatus, but can be used only in fine weather. It is generally established in the upper story of the building, where the air circulates freely. Shelves or racks, on which are placed the pieces of soap to be dried, are fixed in the room eight or ten inches apart, one above the other; this separation has the advantage of accelerating the drying of the soap by putting it in contact with a greater mass of air; the desiccation is more rapid when the temperature of the air is elevated. This mode of drying is the most economical, requiring neither apparatus nor fuel; it is also the most regular and the best for the drying of soaps, and it may be used whenever circumstances will permit. Unfortunately it is, however, subject to the variations of seasons and weather so frequent in our climate. The drying-rooms with warm air have the advantage that they can be used at all seasons. In many factories the drying-room consists of a more or less large room around which shelves provided with trays are disposed and upon which are placed the pieces of soap to be dried. In the middle of the room is a stove heated with wood or coal. The temperature must not be above 80° F.; openings must be made in different parts of the room to permit the air saturated with moisture to escape freely. This arrangement quickly hastens the drying of the soap. A temperature of 80° F. is sufficient to dry in fifteen or twenty hours pieces of olein soap intended to be moulded.

But this mode of drying presents the inconvenience of localizing and causing an unequal distribution of the heat. Some shelves are remote from the source of heat, so that the soap does not dry equally in all parts of the room. And, besides, stoves often smoke, especially when first lighted, and the smoke stains

and blackens the pieces of soap. These inconveniences have obliged some manufacturers to use a

*Drying-room heated by hot air.*—By this system the heat produced by the fuel is completely utilized and the hot air which flows into the room is always pure, without either odor or smoke. What distinguishes this system from all others is that the desiccation of the soap is rather produced by an energetic ventilation, occasioned by the abundance of the hot air continually renewed in the room, than by a high temperature ; and experience proves that in rooms heated by a good stove it requires twenty-five to

Fig. 73.



Drying Room with Warm Air.

A, furnace ; B, furnace door ; C, aperture for the introduction of cold air ; D, smoke-flue ; E, chimney ; F, slide ; G, vent-holes for the escape of the air more or less saturated with the moisture of the room ; H, door by which the trays full of soap are introduced ; J, apertures in the floor for the admission of hot air.

thirty hours to dry the soap, while with a much smaller expense of fuel three times the quantity of soap can be dried in eight or ten hours in a room heated by hot air. Fig. 73 shows a longitudinal section of a drying-room with hot air.



*A* is the furnace in which the fuel is burned for heating the air. Through the door *B* the fuel is placed upon the grate, while cold air is admitted through the aperture *C*. The cold air is heated by circulating round the furnace *A*, and enters the drying-room through apertures in the floor *J*, as indicated by the arrows. The cast-iron pipe *D* conducts the smoke to the chimney *E*; the draft and the temperature of the hot air are regulated by the slide *F*.

The manner of using the drying-room is very simple. After filling the trays with pieces of soap, they are introduced into the room by the door *H*; the door is closed and the fire started. The cold air enters by the aperture *C*, becomes heated by circulating around the furnace, and flows continuously into the room by the apertures *J*, and leaves it saturated with moisture by the vents *G*. The temperature in the room should not exceed 80° to 86° F.

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## CHAPTER XI.

## FABRICATION OF SOAPS.

*Division of Soaps.*

SOAPS may be divided into two principal groups, viz., hard or soda soaps and soft or potash soaps. The first group comprises curd or grain soaps, half-grain soaps and paste soaps. The curd soaps show a characteristic crystalline formation termed "curd" or "grain." They are prepared either by freeing the alkaline oleate from superfluous water and glycerin by the aid of common salt (salting out) or by not completely salting out, but adding only sufficient common salt or using an excess of lye, so that a precipitate of paste is formed from the soap at rest. This latter kind of curd soap can only be prepared by the use of cocoanut or palm-kernel oil in connection with other fats; it is technically known as *curd soap upon a precipitate of paste*, or *deposited curd soap*, while the first variety is termed *curd soap upon sub-lye*. These curd soaps, when again heated upon water or weak lye, are known as *ground curd soaps*.

*Paste soaps* are obtained by simply allowing the soap-paste to congeal. They are prepared with the aid of cocoanut-oil and palm-kernel oil. They contain all the glycerin present in the fat subjected to saponification and a large amount of water. They are either smooth or exhibit but little formation of grain, which, if mottled soaps are to be prepared, can, however, be brought out stronger by the addition of coloring substances such as Frankfort black, ultramarine, colcothar, etc.

*Half-grain soaps* (in Germany known as *Eschereg soap*) can only be prepared with the aid of cocoanut and palm-kernel oil; they exhibit some formation of grain which may also be brought out stronger by the addition of coloring substances. These half-grain soaps are boiled either by the direct or the indirect method.

The first process consists in boiling cocoanut-oil or palm-kernel oil together with other fats, such as palm-oil, bone-fat, fuller's fat, etc., and the second by preparing a grain soap from one or several of the last-named fats, and adding to this a paste soap boiled from cocoanut-oil or palm-kernel oil.

*Potash or soft soaps* are divided into three principal varieties: (1) *transparent soap*, variously colored and brought into commerce under different names, such as oil soap, crown soap, green soap, black soap, glycerin soft soap, etc.; (2) *grained (natural grained) soap*; and (3) an opaque soap of a white or yellowish-white color, designated as *elaidin soft soap*, *silver soap*, etc.

Before entering upon a description of the methods of boiling the most important soaps found in commerce, we will make a few general remarks on the process of boiling.

### *Boiling of Soaps.*

The saponification of fats by the alkalies is in most cases effected by boiling, a lower temperature being generally only used for cocoanut-oil. Mège-Mouries has demonstrated that under certain circumstances other fats can also be saponified at a temperature below the boiling point. If melted tallow is emulsified by shaking with white of egg or gall, and allowed to stand from 3 to 4 hours at a temperature of from 113° to 140° F., it is completely converted into soap at above 140° F. According to Knapp, all fats possess this property even at an ordinary temperature, and without the use of white of egg or a similar substance. By pouring a few drops of caustic soda into a bottle, then allowing it to run out and rinsing with water, every variety of fat brought afterwards into the bottle and shaken with half or equal its volume of water, is readily converted into an emulsion. Solid fats, such as tallow, require a certain degree of heat to render them fluid, and must only be shaken as long as the temperature does not sink below this point, as otherwise they separate in lumps like butter in beating cream. Such emulsions can be diluted at pleasure, and allowed to stand for days without separating into a coherent layer of fat and water, a thick cream only collecting over the thinner milky liquid. Under the micro-



scope such an emulsion appears as a multitude of transparent globules of fat of a very small diameter floating in the clear fluid. The emulsion is, therefore, solely a fine division of the fats similar to the grinding or pulverizing of other substances. By mixing the undiluted emulsion with an excess of caustic lye and allowing the mixture to stand a few hours, the milky condition disappears and the whole separates into clear lye upon which floats a coherent cake of soap. This cake of soap is, according to the strength of the lye used, either hard and brittle or soft and more jelly-like. As regards the rest, the strength of the lye exerts no influence, provided it is not below three per cent. Hence, with finely divided fats saponification takes place with great ease in the cold. Though melted or naturally fluid fats mix in their ordinary condition with lye to an emulsion-like mass, saponification is much more difficult: 100 parts by weight of lard mixed with 400 parts by weight of water and 60 parts by weight of caustic potash were only converted into soap after digesting for two days at a temperature of from  $158^{\circ}$  to  $194^{\circ}$  F. (Chevreul.)

The process of saponification, however, is not even accelerated, as might be expected, by boiling with lye. The fat, to be sure, soon fixes a portion of the alkali, a complete soap being, however, generally only formed by a gradual absorption, sometimes only after boiling several days. The strength of the lye is of great importance for the success of the process, most fats being only saponified with ease by boiling by commencing the operation with weak lyes and gradually increasing their strength. The reason for this different behavior of emulsionized from that of non-emulsionized fats is evidently found in the fact that in using an emulsion the globules of fat offer a number of attacking points to the lye in which they float. By the use of a boiling heat the fat forms a melted, coherent mass which spreads out over the lye, and hence there is not the same intimate mixture of fat and lye, and saponification is effected with greater difficulty. That weaker lyes induce saponification with greater ease than stronger ones is explained by the fact that soaps are insoluble in strong lyes. In modern times quite strong lyes are frequently used for the preliminary boiling, which is, however, only admissible

when the lyes contain much alkaline carbonate. Coconut-oil and palm-kernel oil are, however, exceptions in this respect, they requiring strong caustic lyes for saponification.

The reason why almost all soaps are prepared by boiling, notwithstanding that saponification is not promoted by it, is that the mass formed by the combination of fat and lye requires at least an evaporation of the excess of water to make it into soap, an operation best effected by boiling. Every soap-boiler knows that strong boiling does not promote saponification, and for that reason insists upon a moderate fire in the preparatory boiling.

*Boiling by steam.*—In Germany soaps are generally boiled in open kettles over a naked fire, though almost all large establishments are provided with steam apparatus. They use, however, steam chiefly for melting out the fat from the barrels and for the preparation of lyes, and frequently for the preparatory boiling of special kinds of soap; clear boiling, however, is generally effected over an open fire. The reason for this is that a large quantity of water has to be evaporated from most soaps, which, with the viscosity of the soaps and great depths of the kettles, is possible neither with direct nor indirect steam of ordinary tension. It might be effected with superheated steam, which has been repeatedly recommended for boiling soap, and, it is claimed, is advantageously used for the purpose in France, Italy, and Russia, though thus far it has not been introduced into Germany.

Either direct or indirect steam can be used for boiling soap. In using direct steam it is admitted through a pipe system in the interior of the kettle, and the fat and lye are brought to the boiling point quicker than by means of an open fire. The combination of the fat with the alkali taking place very rapidly, the contents of the kettle may boil over, which can, however, at once be remedied by shutting off the steam. The water produced by the condensation of the direct steam will, as previously remarked, lower the strength of the lyes used, which involves the necessity of employing more concentrated lyes.

The soap-paste obtained by boiling with steam has to be either salted out or further evaporated. For salting out it is best to use a solution of common salt in sub-lye from a former boiling. With the use of dry salt more time is required for the operation,

since the salt falls directly to the bottom of the kettle, where it lies until it dissolves by itself. With an open fire, solution would be more quickly effected by the salt being constantly stirred up by the required agitation of the contents of the kettle, which is omitted by boiling with steam.

For the preparatory boiling or pasting and boiling to grain of soaps which are to be salted out, boiling with steam is the quickest and surest method, the process offering the further advantage of the product showing always a light color, since it cannot be injured by the fire. After drawing off the sub-lye, an open fire is, however, used to prevent the incorporation of unnecessary water which later on would have to be again evaporated. The application of direct steam is in order only for fitting ground curd-soaps, and with the use of lyes of sufficient strength, such soaps can be frequently entirely finished and the salted-out curd be often obtained free from froth, especially by employing caustic soda lyes. The water formed by the condensation of the steam effects here a gradual grinding of the soap.

For boiling with indirect steam the kettle is provided with a serpentine pipe, which runs in several curves along the walls and finally ends outside the kettle. To regulate the admission of steam the serpentine pipe is provided with a cock.

For boiling with indirect steam a higher pressure is required than with direct steam. The mixture of fat and lye is quickly brought to the boiling point, the rising over of the contents of the kettle being prevented by shutting off the steam. The formation of water by the condensation of steam being here avoided, lyes of ordinary strength are used. For soaps which are to be salted out this method is all that can be desired, since, by the lye being constantly kept in a state of uniform ebullition, the boiling proceeds smoothly and regularly.

It is, however, different in boiling half-grain soap, smooth white curd soap (by the indirect method), or soft soap. These are soaps from which a portion of the moisture required for combination has to be evaporated. As long as the soaps are thin, *i. e.*, contain a large amount of water, everything proceeds smoothly, but as soon as the soaps become thick their further evaporation with indirect steam is impossible.



In this country, where the kettles are provided with direct as well as indirect steam (see p. 247), the boiling of resin-grain soaps is effected as follows: After bringing the fat with the required first lye into the kettle, direct steam is admitted or the mixture is brought to boiling by indirect steam. It is then kept in ebullition by direct steam, and lye is added until the paste becomes clear. Salting out is then proceeded with in the usual manner, and the salt being dissolved the direct steam is shut off and boiling with indirect steam continued until a sample of the lye appears perfectly clear and thinly fluid. After the time required for settling the sub-lye is drawn off. The lye required for clear boiling is then brought into the kettle and boiling with indirect steam continued until the soap shows the characteristics of well-boiled curd soap.

Closed apparatus is sometimes used for boiling soap in this country as well as in England. It has the disadvantage that the course of the process cannot be sufficiently watched.

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Before boiling the soap a calculation of the quantities of fat and alkali to be used is necessary, the method of an approximate estimation still much in vogue being by no means a rational one. Frequently the results of the calculations are only approximate, since the different fats and oils contain varying quantities of the different glycerides and of moisture, and not all the constituents of the fats and oils are saponifiable and the lyes of the soap-boiler are impure.

The calculation is effected according to the atomic theory:—

Carbon (C) = 12; hydrogen (H) = 1; oxygen (O) = 16.

It is already known that a molecule of fat containing therein three univalent atoms of acid fixed on the trivalent glyceryl requires three molecules of caustic alkali for decomposition, whereby one molecule of glycerin and three molecules of alkaline oleate are formed; hence each atom of acid contained in fat fixes one molecule of caustic alkali or half a molecule of potassium oxide or sodium oxide.

Stearic acid. $\text{C}_{18}\text{H}_{35}\text{O} \left. \begin{array}{c} \text{H} \end{array} \right\} \text{O}$		Palmitic acid. $\text{C}_{16}\text{H}_{31}\text{O} \left. \begin{array}{c} \text{H} \end{array} \right\} \text{O}$		Oleic acid. $\text{C}_{18}\text{H}_{33}\text{O} \left. \begin{array}{c} \text{H} \end{array} \right\} \text{O}$	
$18\text{C} = 216$ $36\text{H} = 36$ $2\text{O} = 32$		$16\text{C} = 192$ $32\text{H} = 32$ $2\text{O} = 32$		$18\text{C} = 216$ $34\text{H} = 34$ $2\text{O} = 32$	
1 mol. $\text{C}_{18}\text{H}_{36}\text{O}_2 = 284$		1 mol. $\text{C}_{16}\text{H}_{32}\text{O}_2 = 256$		1 mol. $\text{C}_{18}\text{H}_{34}\text{O}_2 = 282$	
Potassium hydroxide.		Sodium hydroxide.		Potassium oxide.	
$\text{K} \left. \begin{array}{c} \text{H} \end{array} \right\} \text{O}$		$\text{Na} \left. \begin{array}{c} \text{H} \end{array} \right\} \text{O}$		$\text{K} \left. \begin{array}{c} \text{K} \end{array} \right\} \text{O}$	
$\text{K} = 39$ $\text{H} = 1$ $\text{O} = 16$		$\text{Na} = 23$ $\text{H} = 1$ $\text{O} = 16$		$\text{K} = 39$ $\text{K} = 39$ $\text{O} = 16$	
1 mol. $\text{KHO} = 56$		1 mol. $\text{NaHO} = 40$		1 mol. $\text{KKO} = 94$	
				$\frac{1}{2}$ mol. $\text{KKO} = 47$	
				1 mol. $\text{Na}_2\text{O} = 62$	
				$\frac{1}{2}$ mol. $\text{Na}_2\text{O} = 31$	

There are required for the saturation of—

284 parts by weight of stearic acid	56 parts by weight of potassium hydroxide.
256     "     "     palmitic acid 60	"     "     sodium hydroxide.
282     "     "     oleic acid 47	"     "     potassium oxide, etc.

Stearin. $(\text{C}_{18}\text{H}_{35}\text{O})_3 \left. \begin{array}{c} \text{C}_3\text{H}_5''' \end{array} \right\} \text{O}_3$		Palmitin. $(\text{C}_{16}\text{H}_{31}\text{O})_3 \left. \begin{array}{c} \text{C}_3\text{H}_5''' \end{array} \right\} \text{O}_3$		Olein. $(\text{C}_{18}\text{H}_{33}\text{O})_3 \left. \begin{array}{c} \text{C}_3\text{H}_5''' \end{array} \right\} \text{O}_3$	
$57\text{C} = 684$ $110\text{H} = 110$ $6\text{O} = 96$		$51\text{C} = 512$ $98\text{H} = 98$ $6\text{O} = 96$		$57\text{C} = 684$ $104\text{H} = 104$ $6\text{O} = 96$	
1 mol. $\text{C}_{57}\text{H}_{110}\text{O}_6 = 890$		1 mol. $\text{C}_{51}\text{H}_{98}\text{O}_6 = 806$		1 mol. $\text{C}_{57}\text{H}_{104}\text{O}_6 = 884$	

Potassium hydroxide.

$\text{K} \left. \begin{array}{c} \text{H} \end{array} \right\} \text{O}$
$3\text{K} = 117$ $3\text{H} = 3$ $3\text{O} = 48$
3 mol. $\text{KHO} = 168$

Sodium hydroxide.

$\text{Na} \left. \begin{array}{c} \text{H} \end{array} \right\} \text{O}$
$3\text{Na} = 69$ $3\text{H} = 3$ $3\text{O} = 48$
3 mol. $\text{NaH} = 120$

Potassium oxide.

$\text{K} \left. \begin{array}{c} \text{K} \end{array} \right\} \text{O}$
$3\text{K} = 117$ $3\text{K} = 117$ $3\text{O} = 48$
3 mol. $\text{K}_2\text{O} = 282$
$1\frac{1}{2}$ mol. $\text{K}_2\text{O} = 141$

Sodium oxide.

$\text{Na} \left. \begin{array}{c} \text{Na} \end{array} \right\} \text{O}$
$3\text{Na} = 69$ $3\text{Na} = 69$ $3\text{O} = 48$
3 mol. $\text{Na}_2\text{O} = 186$
$1\frac{1}{2}$ mol. $\text{Na}_2\text{O} = 93$

For conversion into soap—

890 parts by weight of stearin require 168 parts by weight of potassium							
hydroxide.							
860	"	"	palmitin	"	120	"	"
sodium							
hydroxide.							
884	"	"	olein	"	141	"	"
potassium							
oxide, etc.							

Suppose every oil or fat consists of one molecule each—

Stearin	.	.	.	.	.	$C_{57}H_{110}O_6$	=	890
Palmitin	.	.	.	.	.	$C_{51}H_{98}O_6$	=	806
Olein	.	.	.	.	.	$C_{57}H_{104}O_6$	=	884
<hr/>								
3 molecules of fat = $\frac{C_{165}H_{312}O_{18}}{3} = \frac{2580}{3}$								

Hence 1 molecule of fat =  $C_{55}H_{104}O_3$  = 860 parts by weight, requires 120 parts by weight of sodium hydroxide, or 168 parts by weight of potassium hydroxide, or 93 parts by weight of sodium oxide and 141 parts by weight of potassium oxide.

For the calculation of the quantities of alkali the following general formula, therefore, serves:—

$$\frac{b \times c}{a} : X; X = \text{the quantities of alkali sought,}$$

$$\frac{a \times d}{b} : X; X = \text{the quantity of fat sought,}$$

In which—

- a* is the atomic weight of the acid or the glyceride,
- b* the atomic weight of the alkali,
- c* the quantity of acid or glyceride present, and
- d* the quantity of alkali present.

There are required for—

100 parts of—	Sodium	Potassium	Sodium	Potassium
	hydroxide,	hydroxide,	oxide,	oxide,
	NaHO.	KHO.	Na <sub>2</sub> O.	K <sub>2</sub> O.
	Parts.	Parts.	Parts.	Parts.
Stearic acid . . .	14.08	19.71	10.91	16.55
Palmitic acid . . .	15.62	21.87	12.11	18.36
Oleic acid . . .	14.19	19.85	10.99	16.66
Stearin . . .	13.48	18.87	10.44	15.84
Palmitin . . .	14.88	20.84	11.53	17.49
Olein . . .	13.57	19.00	10.45	15.99
Fat and oil . . .	13.94	19.54	10.81	16.39



There are required for 100 parts of—

Sodium hydroxide, NaHO. Parts.	Potassium hydroxide, KHO. Parts.	Sodium oxide, Na <sub>2</sub> O. Parts.	Potassium oxide, K <sub>2</sub> O. Parts.	
700.0	507.1	916.1	604.2	Stearic acid.
640.0	457.1	825.8	544.7	Palmitic acid.
705.0	503.5	909.7	600.0	Oleic acid.
741.5	535.7	957.0	627.6	Stearin.
671.5	479.7	867.7	571.6	Palmitin.
735.0	530.9	950.5	626.9	Olein.
716.6	511.9	924.7	609.9	Fat and oil.

1 part requires (in round numbers)—

7.17	5.12	9.25	6.1	Fat and oil.
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To facilitate the calculations we give the following tables, which indicate the quantity of fat to be saponified by lye of any desired strength according to Baumé's and Twaddle's degrees :—

*Table of the quantities of fat which a soda lye of Baumé's or Twaddle's degrees requires for saponification.*

Degrees according to Baumé.*	Degrees according to Twaddle.	100 kilogrammes of soda lye		100 liters of soda lye	
		contain per cent. sodium oxide,* Na <sub>2</sub> O.	require kilogrammes of fat.	contain kilogrammes of sodium oxide,* Na <sub>2</sub> O.	require kilogrammes of fat.
1	1.4	0.47	4.35	0.4	4.35
2	2.8	0.93	8.60	0.9	8.60
3	4.4	1.55	14.34	1.6	14.80
4	5.8	2.10	19.42	2.2	20.35
5	7.4	2.60	24.05	2.7	24.97
6	9.0	3.10	29.67	3.2	29.60
7	10.4	3.60	33.30	3.8	35.15
8	12.0	4.10	37.92	4.3	39.75
9	13.4	4.55	42.10	4.9	44.42
10	15.0	5.08	46.99	5.5	50.87
11	16.6	5.67	52.44	6.1	56.42
12	18.2	6.20	57.35	6.8	62.90
13	20.0	6.73	62.25	7.4	68.45
14	21.6	7.30	67.52	8.1	74.92
15	23.2	7.80	72.15	8.7	80.47
16	25.0	8.50	78.62	9.6	88.80
17	26.8	9.18	84.17	10.4	92.87
18	28.4	9.80	90.65	11.2	103.60
19	30.4	10.50	97.12	12.1	111.92
20	32.4	11.14	103.05	12.9	119.32
21	34.2	11.73	108.50	13.7	126.72
22	36.0	12.33	114.05	14.6	135.05
23	38.0	13.00	120.25	15.5	143.37
24	40.0	13.70	126.72	16.4	151.70
25	42.0	14.40	133.20	17.4	160.62
26	44.0	15.18	140.41	18.5	171.12
27	46.2	15.96	147.63	19.6	181.30
28	48.2	16.76	155.03	20.8	192.40
29	50.4	17.55	162.23	22.0	203.50
30	52.6	18.35	169.73	23.0	212.75
31	55.9	19.23	178.61	24.5	226.62
32	57.0	20.00	185.00	25.7	237.72
33	59.4	20.80	192.40	27.0	249.75
34	61.6	21.55	199.34	28.2	260.85
35	64.0	22.35	206.74	29.5	272.87
36	66.4	23.20	214.60	30.9	285.82
37	70.2	24.20	223.85	32.6	301.55
38	71.4	25.17	232.82	34.2	316.55
39	74.0	26.12	241.61	35.9	332.07
40	76.6	27.10	250.67	37.5	346.87
41	79.4	28.10	259.92	39.2	362.60
42	82.0	29.05	268.71	40.0	370.00
43	84.8	30.08	278.24	42.8	395.90
44	87.6	31.00	286.75	44.6	412.55
45	90.6	32.10	296.92	46.6	431.05
46	93.6	33.20	307.10	48.7	448.62
47	96.6	34.40	318.20	51.0	471.75
48	101.2	35.70	330.22	53.5	484.87
49	102.8	36.90	341.32	55.9	517.07
50	106.0	38.00	351.50	58.1	537.42

\* For the specific gravities corresponding to Baumé's and Twaddle's degrees and the quantities of sodium oxide corresponding to the quantities of sodium hydroxide see pages 238 and 240.

*Table of the quantities of fat which a potash lye of Baumé's and Twaddle's degrees requires for saponification.*

Degrees according to Baumé.*	Degrees according to Twaddle.	100 kilogrammes of potash lye contain per cent. potassium oxide,* K <sub>2</sub> O.	100 kilogrammes of potash lye require kilogrammes of fat.	100 liters of potash lye contain kilogrammes of potassium oxide,* K <sub>2</sub> O.	100 liters of potash lye require kilogrammes of fat.
1	1.4	0.70	4.27	0.7	4.27
2	2.8	1.40	8.54	1.4	8.54
3	4.4	2.20	13.42	2.2	13.42
4	5.8	2.90	17.69	3.0	18.30
5	7.4	3.80	23.18	3.9	23.79
6	9.0	4.70	28.67	4.9	29.89
7	10.4	5.40	33.94	5.7	34.77
8	12.0	6.20	37.82	6.6	40.26
9	13.4	6.90	42.09	7.4	45.14
10	15.0	7.70	46.97	8.3	50.63
11	16.6	8.50	51.85	9.2	56.12
12	18.2	9.20	56.12	10.0	61.00
13	20.0	10.10	61.61	11.1	67.71
14	21.6	10.80	65.88	11.9	72.59
15	23.2	11.60	70.76	12.9	78.69
16	25.0	12.40	75.64	14.0	85.40
17	26.8	13.20	80.52	15.0	91.50
18	28.4	13.90	85.79	15.9	96.99
19	30.4	14.80	90.28	17.0	103.70
20	32.4	15.60	95.16	18.1	110.41
21	34.2	16.40	100.04	19.2	117.12
22	36.0	17.20	104.92	20.3	123.63
23	38.0	18.00	109.80	21.4	130.54
24	40.0	18.80	114.68	22.6	137.86
25	42.0	19.60	119.56	23.7	144.57
26	44.0	20.30	123.83	24.8	150.28
27	46.2	21.10	128.71	26.0	158.60
28	48.2	21.90	133.59	27.2	165.92
29	50.4	22.70	138.47	28.4	174.24
30	52.6	23.50	143.35	29.7	181.17
31	54.8	24.20	147.62	30.8	187.88
32	57.0	25.00	152.50	32.1	195.81
33	59.4	25.80	157.38	33.8	205.18
34	61.6	26.70	162.87	34.9	212.89
35	64.0	27.50	167.75	36.3	221.43
36	66.4	28.30	172.63	37.7	229.97
37	69.0	29.30	178.73	39.4	240.34
38	71.4	30.20	184.22	41.0	250.10
39	74.0	31.00	189.10	42.5	259.25
40	76.6	31.80	193.98	44.0	268.40
41	79.4	32.70	199.47	45.7	278.77
42	82.0	33.50	204.35	47.2	287.92
43	84.8	34.40	209.84	49.0	298.90
44	87.6	35.40	215.94	50.9	310.49
45	90.6	36.50	222.65	53.0	323.30
46	93.6	37.50	228.75	54.9	334.89
47	96.6	38.50	234.85	57.1	348.31
48	99.6	39.60	241.56	59.3	361.73
49	102.8	40.60	247.66	61.5	375.15
50	106.0	41.50	253.15	62.5	381.25

\* For the specific gravities corresponding to Baumé's and Twaddle's degrees and the quantities of potassium oxide corresponding to those of potassium hydroxide see pages 238 and 240.



In every chemical equation, hence also in saponification, the product (soap) is equal to the educt—sodium oxide, potassium oxide, water, and fat or oil.

$$\underbrace{1 \text{ molecule stearin} =}_{890 \text{ parts by weight}} \quad \text{and} \quad \underbrace{3 \text{ molecules sodium hydroxide} =}_{(3 \times 40) 120 \text{ parts by weight}} = 1010$$

Give after saponification—

$$\underbrace{3 \text{ molecules sodium stearate} =}_{(3 \times 306) 918 \text{ parts by weight}} \quad \text{and} \quad \underbrace{1 \text{ molecule glycerin} =}_{92 \text{ parts by weight}} = 1010$$

$$\underbrace{1 \text{ molecule fat or oil} =}_{860 \text{ parts by weight}} \quad \text{and} \quad \underbrace{3 \text{ molecules potassium hydroxide} =}_{(3 \times 56) 168 \text{ parts by weight}} = 1028$$

Give after saponification—

$$\underbrace{3 \text{ molecules of sodium stearate, palmitate, and oleate} =}_{936 \text{ parts by weight}} \quad \text{and} \quad \underbrace{1 \text{ molecule glycerin} =}_{92 \text{ parts by weight}} = 1028$$

$$\underbrace{1 \text{ molecule palmitin} =}_{806 \text{ parts by weight}} \quad \text{and} \quad \underbrace{3 \text{ molecules water} =}_{(3 \times 18) 54 \text{ parts by weight}} = 860$$

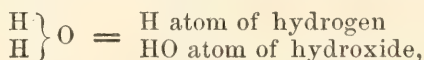
Yield after decomposition—

$$\underbrace{3 \text{ molecules palmitic acid} =}_{(3 \times 256) 768 \text{ parts by weight}} \quad \text{and} \quad \underbrace{1 \text{ molecule glycerin} =}_{92 \text{ parts by weight}} = 860$$

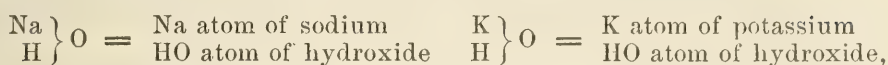
The triglycerides contain—

Stearin . . .	95.35 p. c., remnant of stearic acid, $C_{18}H_{35}O$	} O	} 100.00
4.65 “ “	glycerin, $C_3H_5'''$		
Palmitin . .	94.66 p. c., remnant of palmitic acid $C_{16}H_{31}O$	} O	
5.34 “ “	glycerin, $C_3H_5'''$		
Olein . . .	95.30 p. c., remnant of oleic acid, $C_{18}H_{33}O$	} O	
4.70 “ “	glycerin, $C_3H_5'''$		
Oil or fat . .	95.23 p. c., remnant of fatty acid		
4.77 “ “	glycerin, $C_3H_5'''$		

Now, in the saponification with water, these different remnants divide themselves in the remnants of the water,



or in the remnants of the modified waters of the lyes,



in such a manner that the atom of hydrogen, potassium, or sodium combines with the remnant of the acid, and the atom of hydroxide with the remnant of glycerin = glyceryl  $\text{C}_3\text{H}_5'''$ , so that the original quantity of fat is augmented by the quantity of alkali.

The remnants of acids of the various triglycerides become augmented by—

	Hydrogen.	Sodium.	Potassium.
Stearin . . . .	0.30 p. c.	8.02 p. c.	13.12 p. c.
Palmitin . . . .	0.36 “	8.54 “	14.47 “
Olein . . . .	0.32 “	7.79 “	13.22 “
Oil or fat . . . .	0.34 “	7.91 “	13.42 “

And the remnants of glycerin from—

	Stearin,	Palmitin,	Olein,	Oil or fat,
By hydroxide . . .	5.68 p. c.	6.08 p. c.	5.69 p. c.	5.93 p. c.

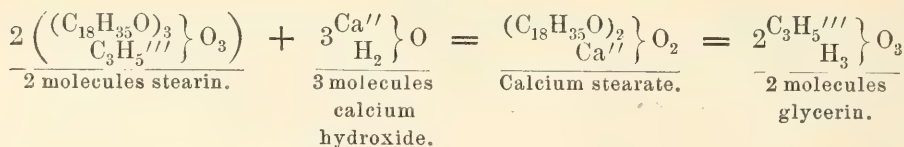
Hence 100 parts of the triglycerides give, after saponification with water, sodium hydroxide or potassium hydroxide—

	Per cent.		Per cent.		Per cent.
Hydrogen stearate	95.73	Sodium stearate	103.37	Potassium stearate	108.47
Glycerin . . .	10.33	Glycerin . . .	10.33	Glycerin . . .	10.33
	<u>106.06</u>		<u>113.70</u>		<u>118.80</u>
Hydrogen palmitate	95.02	Sodium palmitate	103.20	Potassium palmitate	109.13
Glycerin . . .	11.42	Glycerin . . .	11.42	Glycerin . . .	11.42
	<u>106.44</u>		<u>114.62</u>		<u>120.55</u>
Hydrogen oleate	95.63	Sodium oleate	103.09	Potassium oleate	108.52
Glycerin . . .	10.39	Glycerin . . .	10.39	Glycerin . . .	10.39
	<u>106.02</u>		<u>113.48</u>		<u>118.91</u>
Hydrogen sebate	95.57	Sodium sebate	103.14	Potassium sebate	108.65
Glycerin . . .	10.70	Glycerin . . .	10.70	Glycerin . . .	10.70
	<u>106.27</u>		<u>113.84</u>		<u>119.35</u>

The pure dry combinations therefore contain—

	Sodium oxide.	Acid.	Potassium oxide.	Acid.
	Per cent.	Per cent.	Per cent.	Per cent.
Stearate . . .	10.13	89.87	14.59	85.41
Palmitate . . .	10.84	89.16	15.56	84.44
Oleate . . .	10.19	89.81	14.33	85.67
Sebate . . .	10.80	89.20	15.50	84.50
		<u>100.00</u>		<u>100.00</u>

If saponification is effected, as in the manufacture of candles, by means of lime (calcium hydroxide), 2 molecules of triglyceride enter into decomposition with 3 molecules of calcium hydroxide.



100 parts of triglyceride yield—

Calcium stearate . .	104.81 p. c.	Calcium palmitate . . . .	106.07 p. c.
Glycerin . . . .	10.33 “	Glycerin . . . .	11.42 “
	<hr/>		<hr/>
	115.14 “		117.49 “
Calcium oleate . . . .	104.85 p. c.	Calcium sebate . . . .	105.62 p. c.
Glycerin . . . .	10.39 “	Glycerin . . . .	10.70 “
	<hr/>		<hr/>
	115.24 “		116.32 “

There are contained in the—

	Calcium oxide.	Acid.	
Stearate . . . .	9.00 p. c.	91.00 p. c.	} 100.00
Palmitate . . . .	9.19 “	90.81 “	
Oleate . . . .	9.03 “	90.97 “	
Sebate . . . .	9.12 “	90.88 “	

### *Some more Modern Methods for the Manufacture of Soap.*

*Saponification by sulphuretted alkalies.*—Pelouze has shown that the sulphuretted alkalies, used in the same manner as caustic alkalies, possess the property of saponifying fats. At an ordinary temperature saponification takes place in five to ten days, but immediately with the aid of heat. In the latter case sulphide of hydrogen escapes and 1 equivalent of sodium sulphide yields the same quantity of soap as 1 equivalent of anhydrous soda.

In regard to this method of saponification, Dullo remarks that though saponification is quickly and completely effected, he cannot confirm the statement that the disagreeable odor can be removed, except Pelouze possesses a means not published by him. Even when carefully avoiding an excess of sodium sulphide and expelling the sulphide of hydrogen by continued heating, a disagreeable smell remained behind which could not be removed.



J. Laurent raises the following objection to Pelouze's method, which deserves attention. Two factories in Marseilles manufacture daily 44,000 pounds of soap, and if they should adopt this new method, 500 cubic metres (17658.29 cubic feet) of hydrogen sulphide would be daily developed, the injurious influence of which upon the health of the workmen and the hygienic condition of Marseilles would be incalculable.

*Direct saponification of oil fruits.*—Liebreich has patented a process for the direct saponification of copra.\* The copra is comminuted, then saponified with caustic soda lye, and the soap paste separated from the cellulose by means of a centrifugal machine. By salting out the paste a grain-soap is obtained which is so hard that it cannot be cut by any of the usual cutting machines.

Soaps made of cocoanut-oil are difficult to salt out, and when finally separated by the use of too much salt, inclose a considerable quantity of the latter. Soaps formerly prepared, according to Liebreich's process, by the Manufactory of Chemical Products at Charlottenburg, from pure copra or from copra and some olein, showed the above defect of containing a considerable quantity of salt, and made the hands rough. G. Heine, of Charlottenburg, prepares at present, according to Liebreich's process, from copra and a considerable quantity of lard, a stock-soap for the manufacture of fine toilet-soaps, which does not exhibit the above defect, his toilet-soaps being remarkably mild and agreeable in washing.

Liebreich claims as an advantage of his process that it is the only sure method by which a neutral soap can be technically obtained. Why this should be the case we cannot comprehend, and are of the opinion that with the usual methods of salting out and grinding a neutral soap can just as surely be prepared.

*Normal soaps.*—Kluge & Co., of Magdeburg, add to the fitted soaps bicarbonates in order to convert any free caustic alkali present into carbonate. The soaps which Dr. Deite had occasion to examine were mild and yielded a good lather; they showed no reaction with curcuma tincture nor with solution of sublimate, which is due to the fact that sodium bicarbonate does not brown curcuma tincture nor redden solution of sublimate.

\* The pulp of the cocoanut.

## CHAPTER XII.

## HARD SOAPS.

## I. CURD OR GRAIN-SOAPS.

*a. Curd or Grain-Soaps upon Sub-Lye.*

THE boiling of curd-soap upon sub-lye may be divided into three principal operations: The preparatory boiling or pasting, the salting out or separation, and the clear boiling or coction. The object of the first operation is the saponification of the fat, that of the second the separation of the soap from the glycerin and the superfluous water, and that of the third the removal of particles of froth due to incomplete saponification and deposited between the separated curd-soap.

*Old German curd-soap.*—Prior to the introduction of artificial soda, wood-ashes were extensively used for making household soaps, and for hard soaps it was customary to salt out with common salt, either directly or in solution. Where wood is burnt as fuel, and wood-ashes abound and are cheap, or where potash can be procured economically, this process may possess interest and be of useful application, although at present in commercial centres this class of soaps is rarely made. Yet it is necessary, for the reasons stated, to give a description of the process.

The preparation of lye from wood-ashes or potash has been given on page 228.

To convert 1100 pounds of tallow into curd-soap about 138 bushels of wood-ashes have to be lixiviated, but at present potash lyes are generally used. For the above quantity of tallow, 440 pounds of lye of  $10^{\circ}$  B. = 1.07 specific gravity = 7.7 per cent. of potassium oxide, are brought into the kettle, and after warming, the tallow is added. To within a short time of the commencement of ebullition a strong fire is maintained, but it is then

moderated and kept so for five hours. The melted tallow is at once converted into a milky mixture, and as long as lye and fat have not formed a homogeneous combination, the mass boils turbulently in the kettle, without, however, rising. When the mass has been brought to a state of homogeneous mixture, called the "close state" as contradistinguished from the condition in which the soap is granulated and separated from the liquids of the kettle, about 1100 pounds of lye of  $16^{\circ}$  to  $18^{\circ}$  B. = 1.125 to 1.142 specific gravity = 12.40 to 13.90 per cent. potassium oxide, are added in four to five portions at short intervals. The boiling now becomes dense and languid and the mass appears of a yellowish-brown color and runs off the spatula in cohesive, long, translucent strings. *The soap boils to a paste*, and during this process it is necessary to examine whether complete saponification has taken place and there is a correct proportion of alkali to fatty acids. If some of the soap be dropped upon a glass and this sample remains clear for some time, becoming turbid only on cooling, the paste is of the right quality; if, however, the sample becomes turbid in a short time, such turbidity is due either to non-saponified fat distributed in small particles in the paste, or to an excess of lye effecting a separation of solid soap. Non-saponified fat is present if a gray border appears immediately on the edge of the sample, while in the presence of an excess of alkali the sample becomes quickly coated with a whitish film. This defect is readily overcome by an addition of lye or tallow. When the paste is sufficiently clear, and a sample brought in contact with the tongue causes a slight burning sensation, or shows, as it is technically termed, a slight "*touch*," it is vigorously boiled in order to deprive it of a portion of the superfluous water, until it runs in threads from the spatula, which is termed "*spinning of the soap*." As soon as this moment arrives, the separation of the soap from its aqueous solution by means of common salt is commenced, which at the same time effects the transformation of the potash soap into soda soap, the latter process taking place by an exchange of the constituents of the common salt with those of the potash soap, whereby soda soap and potassium chloride are formed.

The salt is added gradually and in small portions, and after



each addition the paste is allowed to boil for a short time in order to enable the operator to watch the full effect of the salt, which does not show itself at once. The first addition of salt effects, as a rule, a liquefaction of the paste. After further additions, the soap coagulates and finally comes as a thick mass to the surface. The separation is due to the soap being insoluble in salt solution and the salt withdrawing water from the soap. The fluid separated from the soap, which is called "sub-lye" or "spent lye," contains, besides the common salt added, the potassium chloride formed by the decomposition and the glycerin formed by the saponification.

Great attention was formerly paid to the operation of salting out, since with an insufficient addition of common salt a portion of the soap remains dissolved in the sub-lye, and with too much salt the soap separates too quickly, so that there is danger of the forming of small lumps which combine with difficulty and hence absorb sub-lye. Soap properly salted out must, when taken hot upon the spatula, adhere to it in soft flakes, boil into laminæ, and a sample, on being pressed with the thumb in the palm of the hand, must not smear, but peel off as a solid, dry chip, or, as it is technically called, "have pressure."

When salting out is finished the fire is removed and the settled sub-lye drawn off or pumped out, or the entire contents of the kettle are transferred into the cooling vat and allowed to rest for the sub-lye to settle. A quantity of the weakest lye is then brought into the empty kettle, and the soap, being carefully removed from the sub-lye in the cooling vat, is added and dissolved to a clear paste, which is again carefully salted out. But little salt is required for this operation, as it only serves for the absorption of water. The next operation, "clear boiling" of the soap, is now proceeded with.

The object of clear boiling is to withdraw the superfluous water from the soap, to saponify the last remnants of fat, and to form the soap into a solid mass free from froth. This operation was generally executed by allowing the soap to boil up high with a quiet, uniform fire, keeping the spatula always ready to prevent boiling over by beating the soap, or, as it is technically called, "checking" or "curbing" the soap. The soap, which was at

first not sufficiently separated from the sub-lye, soon seethes up in small bubbles, which is termed "the soap boils tough." With continued boiling, and the kettle half covered with planks, the water now gradually evaporates, the salt lye becoming more and more concentrated, and by withdrawing more water from the soap the latter contracts and becomes more viscid. Large laminae form on the surface, and the steam developed on the bottom of the kettle makes so much noise in escaping through the thickening soap that in the language of the soap-boiler "the soap talks." The froth, now full of large bubbles, subsides gradually, the soap forms a uniform mass, and no more water being withdrawn from it, clear boiling is finished. On repeating the test with the thumb the soap can be rubbed to a dry, flexible chip. After removing the fire, the soap is allowed to rest a few hours for the sub-lye to settle, and then brought into the frame. In the thickly-fluid soap thus brought into the frame, mottling is readily produced by adhering impurities. As mottled curd-soap was formerly considered especially good, bole elutriated in lye, pyrolusite, etc., was frequently mixed with the soap before bringing it into the frame, in order to make the mottling more pronounced. For smooth soap the thick grain was, however, ground with hot water or weak lye, which renders the soap more liquid, and the adhering impurities pass, on account of their specific gravity, into the sub-lye. The soap absorbing some water by this manipulation, the yield is somewhat increased. One hundred pounds of tallow give about 150 pounds of unground curd-soap, while by grinding the yield may be brought to 156 pounds.

Curd-soaps prepared with potash lye are more delicate and softer than those made directly with soda lye, but they are far more expensive, potash being not only worth twice as much as soda with the same percentage of available substance, but a larger quantity is also required. Suppose both substances were chemically pure, 100 pounds of tallow would require for saponification 28 pounds of chemically pure potassium carbonate, or 21 pounds of chemically pure sodium carbonate.

*Tallow (curd) soap.*—This soap is still much used in the household on account of its great economy in washing, and in the textile industries on account of its great unctuousness and other

good qualities. Besides, tallow is the best raw material for the fabrication of curd-soap and gives the greatest yield, 100 pounds giving from 150 to 160 pounds of soap, according to whether it is more or less ground.

The lye required for the fabrication of this soap is prepared from high-graded calcined soda set with 50 to 60 per cent. of lime. It is still better to use caustic soda lyes prepared by dissolving high-grade caustic soda in water, such lye having the advantage that in salting out no sodium carbonate—of which lyes prepared from calcined soda contain always more or less—passes into the sub-lye, and, as is frequently the case, is lost. Sodium carbonate in lyes, as previously mentioned, does not contribute to saponification, and as it is only mechanically mixed with the soap and deposited between the soap-atoms, renders the soap more liquid. In salting out, the sodium carbonate in the paste, not being chemically fixed, is carried into the sub-lye.

The preparation of tallow curd-soap by means of soda lye is much the same as that of old German curd-soap, though at the present time the soap is generally boiled upon one water, except where the impurity of the tallow renders a more frequent renewal of the lye necessary.

The boiling process is generally carried on as follows: The tallow is brought into the kettle, and as it can only be completely saponified with weak and more caustic lye, tank-lye of  $8^{\circ}$  to  $10^{\circ}$  B. (about one-quarter of the entire quantity required for saponification) is added. With a slow fire, the lye and tallow immediately form an emulsion and combine to a paste shortly after the boiling point is reached. This combination, and consequent complete fixation of the alkali by the fatty acids, are recognized by a sample brought in contact with the tongue, showing no longer a biting, but a more sweetish taste. However, if the mass still shows some sharpness, the complete fixation of the alkali must be effected by continuing the boiling.

After a combination of the lye and tallow has been effected, the second portion of lye is added, for which one of  $12^{\circ}$  to  $13^{\circ}$  B. will best serve, and the mass thoroughly boiled. The third and fourth portions of lye of  $12^{\circ}$  to  $13^{\circ}$  B. are then successively added



in the same manner, and the boiling continued until there is a clear thickly fluid mass in the kettle.

If it should happen that by having added the lye too quickly a paste is obtained which, though showing sharpness when brought in contact with the tongue, has a turbid and dull appearance, some very weak lye has to be added with slow boiling, when the sharpness will disappear and the paste become clearer. An addition of water or weak lye is also of advantage, when by the use of too strong lye ( $18^{\circ}$  or  $20^{\circ}$  B.) the combination has been destroyed so that the lye runs separately from the spatula. We would further remark that the use of lyes too low in lime can only be injurious, since they not only combine with difficulty with the tallow, but the large quantity of sodium carbonate always contained in such lyes is liberated by salting out and passes into the sub-lye.

The saponification of the tallow may be considered complete when a sample of the paste brought upon a glass plate shows immediately a narrow whitish border (lye-rings); remains clear for some time, becomes turbid only on cooling, and when brought in contact with the tongue, gives a perceptible burning sensation (touch).

To deprive the paste of a portion of its superfluous water, it is now thoroughly boiled until it runs in cohesive threads from the spatula. It is then separated from its aqueous solution, which is generally effected by means of common salt. For this purpose salt in small portions is added to the soap paste by which it is soon rendered more liquid. The separation of the soap from the sub-lye is then effected by the gradual addition of more salt. This separation is based upon the fact, that the soap is insoluble in strong salt solution, and that the salt withdraws water from the soap.

This salting out is best effected by keeping the soap at a gentle ebullition. Soap correctly salted out should boil into laminae, appear in white flakes upon the spatula, and stand the test of pressure described on p. 302. The sub-lye should run off clear from the spatula, and show not a burning, but a more salty taste. We would further remark that less salt is required for the separation of soap prepared with soda lye than for that with potash

lye, the quantity of salt necessary for the decomposition of the potash soap not being required.

Salting out being completed the fire is removed for the time being to allow the soap to quiet down. A portion of the sub-lye is then pumped off and the operation of clear boiling commenced, which is effected in the same manner as described for old German curd-soap.

Many manufacturers prepare their curd-soap without boiling it clear upon sub-lye. The soap paste is only boiled clear and free from froth, and then carefully separated with salt or strong salt water so that no froth is formed, whereby a grain free from froth is obtained in the kettle. Curd-soap thus prepared gives a greater yield, but remains always very soluble, and when cut into bars frequently dries crooked. Such soaps can, however, be still more quickly and better prepared by means of steam. By the violent motion of the mass, produced by the admission of steam under strong pressure, the tallow is brought into more intimate contact with the lye and saponification is more readily effected, so that a large quantity of tallow is in a short time converted into a clear paste free from froth.

For the fabrication of tallow (curd) soap with caustic soda lyes the process of boiling is the same as above. For pasting, caustic soda lye of  $10^{\circ}$  to  $14^{\circ}$  B. is used. If in working old, somewhat rancid tallow, which contains, as is well known, more free fatty acids, the soap is rendered too caustic, which is indicated by thick and viscid boiling, some salt water of  $20^{\circ}$  B. is carefully added to remove this viscosity, or some soda solution where salt water may be too strong and exert an injurious effect upon the combination. By boiling old, rancid fats upon pure caustic soda lyes, defective curd-soaps are obtained, which is, however, also the case when fresh fats are boiled with lyes containing much alkaline carbonate.

*Marbling or mottling of tallow (curd)-soap.*—When the operation of clear boiling is finished, the fire is removed and the sub-lye allowed to settle. If the soap is to be marbled, the curd is either at once brought into the frame or first rendered somewhat more liquid by crutching in some hot water or weak lye, care being had not to work in any of the sub-lye. The soap being

thoroughly crutched, the filled frames are covered with boards to permit the soap to become thoroughly heated and allowed to stand quietly. The soap becoming heated crystallization takes place, and, as it is technically termed, "grain" and "flux" (Fluss) are formed. The grain is the crystalline portion of the soap and incloses the non-crystalline (the flux), all the impurities and coloring substances from the tallow, soda, etc., contained in the soap passing into the flux. Such soap, when cut, shows a more or less marbled appearance, and is therefore called marbled-curd soap. To make the marbling more prominent, bole elutriated in lye, pyrolusite, Frankfort-black, ultramarine, etc., are added towards the end of the boiling. These coloring substances being mechanically mixed with the soap deposit themselves in the flux.

The marble in curd-soaps is allowed to form either spontaneously or is drawn into "sheaves and flowers," *i. e.*, after the process of formation is complete in the soap a different design is given to it by artificial manipulation, the design resembling either sheaves closely ranged alongside each other or flowers. The following is the process of producing sheaves :—

When the soap in the frame has become sufficiently heated and set, which is recognized by the steam penetrating through the cracks between the boards, and shows the requisite fluidity, which otherwise has to be effected by crutching in some hot water, or weak lye, or by adding several pounds of cocoanut-oil, the process of producing sheaves is commenced after allowing the soap to somewhat cool off.

A round iron rod, provided on top with a handle and below with a small button, is pushed into the soap down to the bottom. With this rod straight lines, as close to each other as possible, are first drawn the length of the frame and then the width. The closer and more uniform these lines are drawn the more beautiful the sheaves will be. Flowers are produced in the same manner; flower-like designs being formed by skilful manipulation and twisting of the iron rod.

Simple mottling, or sheaves and flowers, can, of course, be only produced when the soap is sufficiently hot, crystallization not taking place in soap brought too cold into the frame.

Smooth white-curd soap is obtained either by crutching the



soap in the frame, of course without the addition of coloring matter, until it is observed to congeal, whereby crystallization is prevented and the small quantities of impurities uniformly distributed, or the thick grain in the kettle is sufficiently ground.

*Grinding of soap.*—The object of grinding is (1) to prevent the formation of marbling, (2) to purify the soap if necessary, and (3) to increase the yield by the absorption of water. The operation is performed by two different methods, either “*from above*” or “*from below*,” but is always carried on with water or weak lye.

Grinding from above is generally executed by crutching into the thick curd-soap in the kettle enough hot water or weak lye to sufficiently dissolve and liquefy the soap grains. During this manipulation soap which is too sharp, *i. e.*, contains an excess of alkali, may, if necessary, be somewhat improved by grinding with water, by which the alkali is withdrawn into the sub-lye, or some causticity may be imparted to the soap by grinding with strong lye. Soap of right constitution is generally ground with weak lye ( $3^{\circ}$  to  $4^{\circ}$  B.).

After grinding the soap the kettle is covered with the lid and the soap allowed to stand a few hours for the impurities to pass into the sub-lye.

Grinding from below is effected by pumping out or drawing off the sub-lye, then bringing the required quantity of weak lye, or water with an addition of salt to prevent the complete formation of paste, into the kettle, and effecting the solution and liquefaction of the soap grains by vigorous crutching or gentle boiling. Grinding from below is only necessary with very impure materials, and is but little practised at the present time.

*Utilization of sub-lye.*—The sub-lye from curd-soaps contains common salt, the excess of alkalies used, the glycerin contained in the fats, frequently more or less soap, and the impurities from the fats and the lye. Though, as will be seen from the preceding, it contains valuable substances, it is seldom utilized by soap-boilers and generally allowed to run off, and where facilities for carrying it off are limited causes frequently great inconvenience. Usually an attempt is made to utilize the alkali contained in it by adding fat and boiling. The lyes which finally remain are

seldom clear and thinly fluid, but generally thick and congeal to a jelly-like mass. The soap-boiler who is forced to allow such lyes to run off hot in order to obtain room for a new boiling, loses many pounds of soap contained in them.

In many factories an attempt is made to utilize the sub-lye by evaporating it and selling it as so-called "fulling extract." Evaporation is generally effected over an open fire, though indirect steam is preferable. With an open fire constant stirring is absolutely necessary to keep the bottom of the kettle free, which otherwise would suffer injury and soon wear out. Evaporation is continued until a sample congeals to a jelly interspersed with small crystals of soda and salt. Deep soap-kettles are not well adapted for the operation, the evaporating surface being too small and stirring difficult and almost impossible the moment the separation of crystals commences. Shallow pans can be highly recommended for the purpose, though they take up much space, of which there is seldom an abundance in soap-factories. In many factories the operation is carried on by evaporating the lye in a soap-kettle to the commencement of the separation of crystals and finishing in shallow pans.

Independent of the profit gained by selling the fulling extract, this evaporation of the lyes has the further advantage of drawing attention to the immense quantity of soap contained in them. This content of soap varies with different fats, and, besides, depends on the manner of salting out, the lyes containing more soap if salting out has been effected by an excess of lye than by common salt. However, not every thick, jelly-like sub-lye contains much soap, bone fat, for instance, yielding frequently such thick sub-lye, which is due more to glue than to soap. The jelly-like sub-lye from sulphur olive-oil is due to vegetable albumen.

After boiling the sub-lye for some time a froth forms on the sides of the kettle; this is removed and added in boiling resin grain-soaps.—Sub-lyes from resin grain-soaps are difficult to evaporate, they foaming very much at first and must be kept down by stirring.—To lyes containing very little alkali, which is readily recognized towards the end of evaporation, a few pounds of soda are added.

The finished fulling extract is generally of a dark-brown color,

and is drawn off into old iron vessels, kettles, lye-kettles, etc., to cool, to be filled into barrels when an order is received.

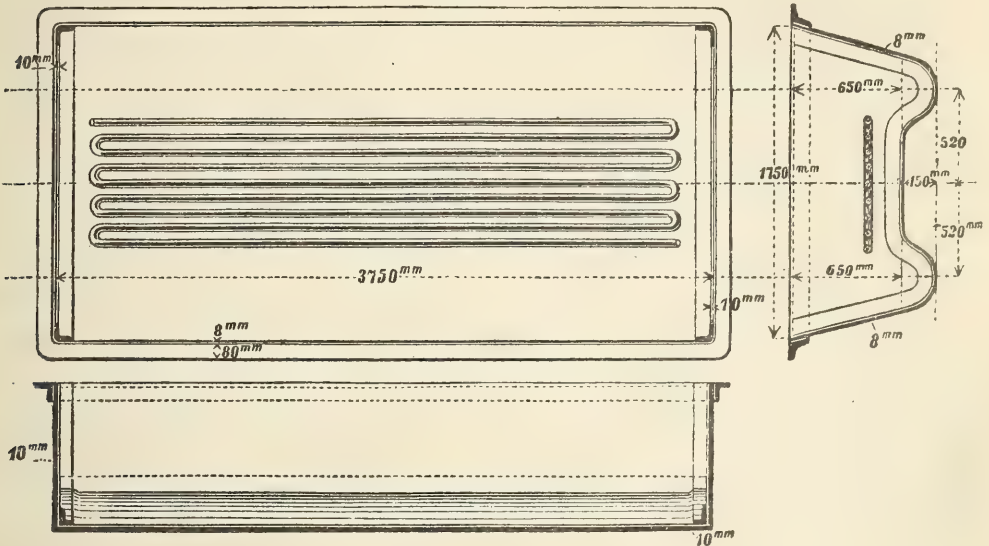
It has been previously mentioned that on account of the great content of common salt the regaining of glycerin from the sub-lye is a difficult matter. On treating glycerin with animal charcoal only a very small portion of the salt is removed and but little of it is carried over in distilling with superheated steam. These evils have for a long time prevented the utilization of sub-lyes for the recovery of glycerin, though many experiments have been made in this direction, however, without success. In 1880 and 1881, when crude glycerin was scarce, many patents were taken out for the purpose, one of which yields excellent results when crude glycerin is bringing a fair price. This is the process of recovering the glycerin by osmose, patented by H. Flemming, of Kalk. The reason why this process has not been generally introduced is partly due to a reduction in the price of glycerin and to the content of glycerin in the sub-lyes varying so much as to make its recovery in many cases unprofitable. In six different sub-lyes, Flemming found, for instance, 7.80, 6.70, 5.50, 4.75, 2.85, and 0.92 per cent. of glycerin. This variation in the content of glycerin is readily explained by the difference in the raw materials, and the soap-boiler who uses them can judge which lyes are suitable for the recovery of glycerin, and exclude those not likely to prove profitable.

To obtain the glycerin by osmose, the sub-lyes have to be evaporated and freed as much as possible from the salts contained in them. For evaporating it is best to use a shallow pan, as shown in Fig. 74, which allows of a convenient removal of the separated salts, or Leon Droux's apparatus with revolving cylinder, shown in Fig. 75. It consists of the metal cylinder *AB*, with the hollow axles *SS*, through which the steam is admitted and later on discharged. The heating surface of the cylinder is considerably augmented by a number of ribs on its circumference. The cylinder dips into the fluid to be evaporated. By an arrangement, as seen in the illustration, the cylinder is slowly revolved, and with each revolution is covered with a thin layer of fluid which readily evaporates even at a comparatively low temperature. The condensed water runs off at *Z*. The salts form an



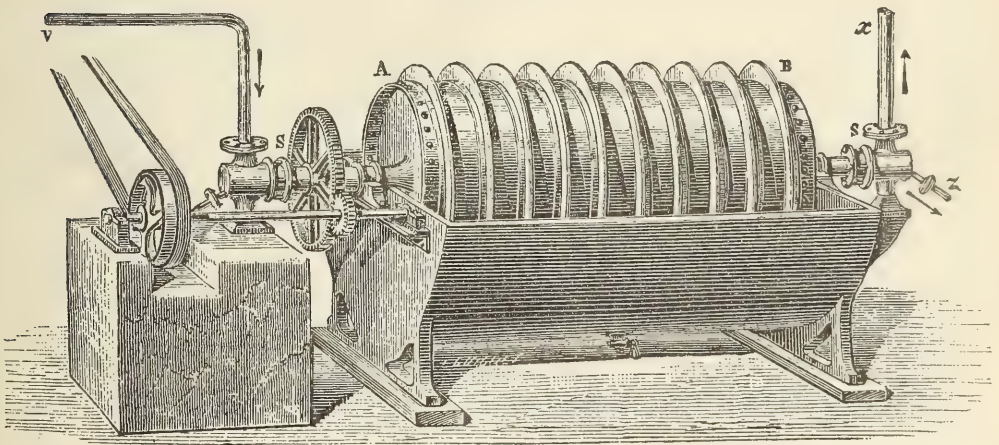
abundant deposit on the surface of the cylinder, and can be readily removed by means of a hammer or by dipping into a less concentrated liquid.

Fig. 74.



In a Marseilles factory where 12 such cylinders, each  $7\frac{1}{4}$  feet long and  $2\frac{3}{4}$  feet in diameter, are employed, more than 11,000 gallons can be evaporated daily.

Fig. 75.



The lye is evaporated to  $30^{\circ}$  B. and then allowed to rest for some time. After cooling a portion of the chlorine combinations and of the sulphates separates out, the liquid, however, retaining

sufficient sulphur combinations to render the recovery of the glycerin impossible. The sulphur combinations are, therefore, oxidized by an addition of sulphuric acid and the introduction of hot air. The liquid is then again evaporated, this time to from  $35^{\circ}$  to  $36^{\circ}$  B., the product being a strongly-colored, syrupy liquid.

Instead of neutralizing the original lye with sulphuric acid, evaporation can be first proceeded with to a certain degree, this method offering the advantage that a considerable portion of the soda and common salt contained in the sub-lye can be recovered.

As the content of common salt is the chief hindrance to the recovery of glycerin from sub-lyes, Jaffé and Darmstädter have recommended the use of Glauber's salt instead of common salt for salting out, as it can be almost completely removed by evaporating the lyes, and, besides, does not pass over with the vapors in distilling with superheated steam. It has been previously mentioned that other salts, and among them Glauber's salt, possess the property of separating the soap from the aqueous solutions, but in a less degree than common salt. The separation by means of Glauber's salt being not a complete one, the sub-lyes contain larger or smaller quantities of soap, and the process has the further disadvantage of light soaps frequently becoming dark on storing.

Albert Domeyer and Otto C. Hagemann, of London, England, have invented and patented under date of October 4, 1887, the following process for recovering glycerin and other products from soap lyes: The spent lyes are collected in a suitable tank, whereupon there is added to them a small proportion of caustic lime, baryta, alumina, or any earthy or metallic oxide capable of combining with the soapy and fatty matters contained in the lye. In ordinary cases it will suffice to add about two pounds of lime to about 100 gallons of lye. The lime, or its above-mentioned equivalents, produces an insoluble precipitate, which quickly settles to the bottom of the tank, and thus the lye is purified from a large proportion of the fatty matters it contained. The lye is next boiled down to the salting point, *i. e.*, it is concentrated until it is saturated with salt. This eliminates a considerable quantity of the water, reduces the bulk of the lye, and brings it into the

best condition for subsequent treatment. The lye is now drawn off into another tank and mixed with an insoluble silicate, such as silicate of alumina (kaolin), or silicate of magnesia, glass-flour, or finely divided silica, the mixing being preferably effected while blowing air through the lye. A good proportion is the addition of about  $1\frac{1}{2}$  pounds of kaolin to 60 gallons of lye. An excess of hydrochloric or other acid is then added and the blowing in of air continued for some time after the acid has been added, allowing the resultant precipitate to settle, whereupon the clear lye is drawn off. By an excess of hydrochloric acid is meant such a quantity of acid as will show from one to one and one-half per. cent. of free acid to the original bulk of the lye. The exact quantity to be added depends, necessarily, upon the fatty and resinous matter contained in the lye. By the addition of the acid, the fatty and resinous constituents of the lye are rendered insoluble, and, being liberated from the solution in the presence of the finely divided kaolin, combine mechanically with the same, forming a heavy precipitate, which quickly subsides, leaving the lye clear and bright.

It will be seen from the above statement that the object of the kaolin or its equivalent is, to a certain extent, of a mechanical character, namely, to mechanically combine with the insoluble resinous matter, causing the same to fall to the bottom. Any matter that will perform this function may be regarded as equivalent to the kaolin; among others a small proportion of light petroleum or other oil may be used. In this case the petroleum rises to the surface of the lye and brings with it the separated fatty and resinous acids which can be conveniently skimmed off. It requires, however, considerable agitation to render this process available, and even then the separation is not as good and clear as by the kaolin method. Soda, either caustic or carbonate, is now added to the lye in such proportion as to render the liquid faintly alkaline. The lye is then heated to about  $176^{\circ}$  F., the resulting precipitate allowed to settle, and the clear lye drawn off. The object of adding the soda is to purify the lye from albuminous matters, which coagulate and fall to the bottom. Instead of soda any other suitable alkali may be added to the lye. The lye is finally heated to about  $302^{\circ}$  F., whereby more water is evap-



orated, causing the salt which is carried in the lye to crystallize. This salt may be washed and used over again in the manufacture of soap. The lye, purified from soapy and fatty matters by the first operation—namely, the addition of lime, from fatty acids and resinous acids by the third operation, namely, the addition of kaolin, and from albuminous matters by the fourth operation, namely, the addition of soda—is thus by the final evaporating process concentrated, so as to eliminate the remainder of the water and most of the salt, giving as a final product crude glycerin, which is fit for distillation without any other treatment.

In many instances the first operation—namely, that of the adding of lime—is not essential, as the third operation—namely, the addition of acid and kaolin—will also suffice by itself to eliminate the fatty and soapy matter; but this requires more acid and renders the process more expensive. If the spent lye is at once treated with lime, it must nearly always be done in a thin state, *i. e.*, before it is boiled down to the salting point. This in most cases is necessary on account of the difficulty experienced in boiling down a soapy lye, from its tendency to froth and boil over. By adding the lime this difficulty is overcome, and the second process of boiling down is rendered practicable; yet, of course by omitting the first and second processes and starting on the spent lye with the hydrochloric acid and kaolin, the same result is arrived at, but at greater expense and less convenience.

As to the third process—that of adding kaolin and an excess of hydrochloric acid—it may be stated that, although other suitable acid may be used, hydrochloric acid is by far the best, because it produces chloride of sodium (common salt), which, when recovered, can be used over again indefinitely in the manufacture of soap.

As to the fourth operation—purifying the lye from albuminous matters—it is necessary to have the lye neutral in order to get the best results, and accordingly it is neutralized with carbonate or caustic soda or other alkali, which, in conjunction with the hydrochloric acid used in operation No. 3, helps to form chloride of sodium.

*Marseilles soap (olive-oil curd-soap).*—This old and celebrated soap, also called castile or Venetian soap, was originally prepared

from olive-oil upon sub-lye, though at present the olive-oil is frequently mixed with other oils. The manufacture of this soap, especially valuable in dyeing silk and Turkish red, has been carried on in Marseilles for centuries, and at the present time has assumed immense proportions.

The olive-oil required for the fabrication of the soap is brought to Marseilles partly from Provence and partly from Spain, Italy, Tunis, and the Levant. Oils of the second pressure only are used; those of the first pressure, besides being consumed as table oils and therefore too expensive, do not contain a sufficient quantity of solid fatty acids. The oils from Provence, Spain, and Naples yield the most solid soaps. In more modern times the manufacturers have commenced to mix the olive-oil with cheaper oils, such as sesame, poppy-seed, rape-seed, and cotton-seed oils, and in Marseilles especially, peanut-oil, in the proportion of 1 to 3 and 1 to 2.

Barilla, a natural soda obtained by the incineration of maritime plants, was formerly used for the preparation of the soap. However, large factories for the manufacture of artificial soda from sea-salt having been established in Marseilles, calcined soda is now chiefly used, as well as caustic soda, and sometimes crude soda.

There are a number of large soap-factories in Marseilles which, as a rule, are excellently arranged and mostly for steam, each factory having generally three to five kettles of large dimensions. The kettles are generally provided with a curb of brick and copper bottom, upon which rests the steam-coil, and on the lowest point of which is a pipe with stop-cock for the discharge of the sub-lye, etc. The oil and required lye are brought into the kettles from reservoirs situated in their immediate neighborhood. The preparation of lye is also carried on in the most advantageous manner, there being a battery of four lye-kettles for each soap-kettle, which are provided with all the modern improvements. The soap is run into the low, but large, soap-frames from the higher situated kettles by means of gutters.

Two varieties of soap of different qualities, marbled and smooth white soap, are chiefly brought into commerce from Marseilles.

The operation of making the soap is divided into the following different parts :—

1. Pasting (*empâtage*).
2. Separation (*relargage*).
3. Hardening (*trempage*).
4. Clear boiling (*coction*).
5. Marbling or mottling (*madrage*).
6. Framing (*colage*).

The object of the first operation is the formation of paste, and for this purpose it is best to use lyes of  $10^{\circ}$  to  $15^{\circ}$  B.

The execution of this operation varies with different manufacturers, some bringing the oil with about one-third part by weight of the same lye of  $10^{\circ}$  B. into the kettle and effecting a union between lye and oil by thorough crutching or by means of a stirring apparatus. Others, however, bring first one-third of the lye of  $10^{\circ}$  to  $11^{\circ}$  B. into the kettle, and after heating by means of steam or over an open fire, add the oil. The same object of establishing a union between lye and oil being accomplished by both methods, this difference in the manipulation is of but little consequence.

After the combination between lye and oil is effected, which is promoted by further heating and stirring, the mass soon reaches the boiling point. If boiling is conducted carefully and with a slow fire, the froth formed in the commencement of ebullition disappears quickly and the mass in a short time acquires a uniform appearance, a sample taken upon the spatula showing neither oil nor lye. Lye of  $15^{\circ}$  B. is now added and the fire increased or more steam admitted. With slow boiling and a further gradual addition of lye of  $15^{\circ}$  B., saponification progresses more and more, the paste becoming thicker and shorter. After fitting the paste with some lye of  $20^{\circ}$  B., whereby a slight excess of alkali (which is indicated by the burning sensation produced by a cold sample upon the tongue) is not injurious, it will have become thick and quite solid. In Marseilles the lye used is generally calculated in liters, and for pasting 2200 pounds of oil, about 700 liters of lye of  $10^{\circ}$  B., 300 liters of  $15^{\circ}$  B., and 100 liters of  $20^{\circ}$  B. are required.

The fire is now withdrawn or the steam shut off and the paste



intended for marbled soap colored. For this purpose a few pounds of ferrous sulphate are mixed with the paste, whereby iron soap is formed, and later on some sodium sulphide (obtained by dissolving flowers of sulphur in strong boiling lye) is added to the separating lye, which decomposes the iron soap and converts it into black ferrous sulphide. This imparts to the soap the old style of greenish-black marbling so much liked. Some manufacturers color their soap by adding ultramarine and ferrous sulphate dissolved in water and boiling for a short time.

To separate the soap from its aqueous solution the second operation, namely, salting out the soap-paste, is now proceeded with. This is, however, not effected by means of pure common salt, but with lye of  $20^{\circ}$  to  $30^{\circ}$  B., containing much common salt, which is added to the paste, with constant vigorous crutching, until the soap shows a granular appearance, which indicates the end of the operation. In most Marseilles factories thoroughly clarified strong sub-lye from a previous clear boiling is used, though concentrated salt lye freshly prepared would be preferable. After allowing the soap to rest for three to four hours, the sub-lye is drawn off by means of a pipe immediately above the bottom of the kettle.

The sub-lye having been drawn off, more salt lye of  $20^{\circ}$  B. (1550 to 1750 pounds of lye to 4400 pounds of soap) is added to the soap in the kettle. A moderate fire is then started or steam introduced and the hot soap thoroughly crutched. By this, the third operation, the soap is somewhat more hardened. It is then allowed to rest a few hours, when the sub-lye is drawn off and the fourth operation—clear boiling—is commenced, whereby a complete saturation of the soap with alkali is effected.

For this purpose about 1100 pounds of fresh, pure lye of  $22^{\circ}$  to  $25^{\circ}$  B. for 4400 pounds of soap are brought into the kettle, and, after starting the fire, allowed to boil for a few hours. The grain being insoluble in the strong lye, contracts more and more, thereby absorbing alkali and yielding up superfluous water. When the soap is completely saturated with alkali, the froth previously covering it will have almost entirely disappeared. A sample of the grain thus obtained can be rubbed to a dry, soft chip and will show good pressure.

The fire is now removed and the whole vigorously crutched through in order to uniformly distribute in the soap the coloring substance, ferrous sulphide and undecomposed iron soap, which on account of their greater specific gravity have settled on the bottom of the kettle.

The soap obtained by these operations is slightly ground with hot water and then brought into wooden frames which are well covered. Crystallization soon takes place in the soap. The non-crystalline portion, which contains the added coloring substances, is inclosed by the white crystalline portion, thus giving the soap its marbled appearance.

After the soap is cut the ferrous sulphide in the marbling absorbs oxygen from the air and is converted into ferrous hydrate and sulphuric acid. The sulphuric acid enters at once into a combination with the alkali of the soap, while the ferrous hydrate is separated as a yellow substance; for this reason the blue marbling of Marseilles soap soon turns yellow after coming in contact with the air.

The cut soap is brought into thoroughly ventilated drying chambers and exposed to a temperature of from  $90.5^{\circ}$  to  $95^{\circ}$  F. When sufficiently solid it is pressed or stamped and is then ready for sale.

For smooth white soap the operation of marbling and the addition of ferrous sulphate, etc., are, of course, omitted. The grain obtained by clear boiling undergoes, however, another operation, viz., grinding.

The sub-lye obtained from clear boiling, as well as that from salting out and hardening, is conducted into a reservoir and used for salting out a fresh boiling.

The object of grinding is to free the soap from adhering alkali and other impurities. It is executed by adding to the grain in the kettle, after discharging the sub-lye, about 440 pounds of lye of  $6^{\circ}$  to  $7^{\circ}$  B. and heating the mass, with thorough crutching. During this manipulation the grain softens, spreads, and finally becomes liquid. When this is the case, and the mass boils slowly, lye of  $3^{\circ}$  to  $4^{\circ}$  B. is added until the grain has contracted so far as to boil in clear, irregular laminæ, and a sample, when pressed in the palm of the hand, "wets," *i. e.*, leaves moisture

behind, and the mass, on crutching, appears dark and pasty. When grinding is completed the fire is withdrawn and the kettle covered warm. After standing 24 to 36 hours, the ladling out of the soap is commenced. The slight froth is first carefully removed from the surface, and the soap, which is of a honey-yellow color, is then ladled into the wooden frames, care being had when coming towards the bottom of the kettle not to dip into the dark paste. The light soap in the frames is then thoroughly crutched to make it as homogeneous as possible.

In Marseilles it is customary to beat the cooled soap with broad pieces of wood to prevent the formation of hollow spaces in the interior. After standing a few days the soap is cut and brought into the drying-room.

The dark paste remaining in the kettle contains all the impurities and the excess of alkali. It is separated by adding strong, salty sub-lye, and the grain thus obtained is used for marbled soap. White Marseilles soap thus prepared contains about 52 per cent. of fatty acid and is one of the best soaps for industrial and household purposes.

One hundred parts of olive-oil give on an average 155 to 158 parts of Marseilles soap.

It cannot be denied that the reputation of Marseilles soap has lately suffered, which is due not only to stronger grinding, but also to an augmentation of weight by filling with heavy spar, talc, etc., as shown by the following analyses:—

According to Ure, 1 and 2; Braconnot, 4; Thenard, 5; Bolley, 3, 6, and 7.	1	2	3	4	5	6	7
	MARSEILLES SOAPS.						
	Curd-soaps.			Ground-soaps.			
	Marbled.	White.	Marbled.	White.	White.	Marbled.	White.
	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>
Water . . .	10.15	14.5	15.50	21.36	45.2	23.94	27.45
Fatty acids . .	76.00	76.5	72.00	68.40	50.2	67.16	62.80
Sodium oxide—							
Fixed . .	8.65	9.0	8.00	10.24	4.6	7.82	9.15
Free . . .	0.25	—	0.45	—	—	—	0.20
Foreign salts, residue	} 4.95	—	4.05	—	—	1.08	0.40



*Marseilles soap from olive-oil fatty acid.*—In modern times, as previously mentioned, the glycerin is frequently extracted from neutral fats, such as olive-oil, palm-oil, tallow, etc., before working them into soaps. For this purpose the fats are saponified in autoclaves, and the fatty acids obtained frequently distilled with superheated steam in order to obtain them white. Such a refined fatty acid obtained from olive-oil is of a lardaceous consistency and a pale-yellow color, and is frequently used in Germany for the manufacture of Marseilles soap. The boiling process differs somewhat from the ordinary one and is briefly as follows:—

The required lye of  $12^{\circ}$  to  $15^{\circ}$  B. is brought into the kettle, and when boiling a small quantity of the fatty acid is added with thorough crutching. After the solution of the fatty acid in the boiling lye, other portions of the fatty acid are gradually added, care being had to see that one portion is entirely absorbed by the lye before adding the next. This is continued until a clear, thick paste is formed. When a sample brought in contact with the tongue shows a slight touch, the addition of fatty acid is interrupted. After allowing the paste to boil a short time it is finally salted out with lye of  $30^{\circ}$  B. The grain is then boiled clear in the same manner as tallow (curd) soap, and after removing the sub-lye thoroughly ground and fitted. After standing overnight in the covered kettle, the soap is ladled into small shallow frames and crutched cold. Marseilles soap thus manufactured is very white and solid.

*Palm-oil (curd) soap.*—Palm-oil contains a large amount of palmitin, and is readily saponified either by itself or in connection with other fats, such as tallow, bone-fat, olein, etc., the soaps thus prepared being much liked in the various branches of the textile industry as well as in the household. Comparatively little palm-oil is used in a crude state, it being generally bleached before use to remove the coloring substance and other impurities.

Pure palm-oil soap is prepared in the following manner: A determined quantity of bleached palm-oil and a portion of the required lye of  $14^{\circ}$  B. (about 550 pounds of lye to 1000 pounds of oil) are brought into the kettle. With a slow fire and crutching an emulsion is soon formed, and, when ebullition is reached, the fat will intimately combine with the lye. When this is the

case more lye of  $14^{\circ}$  B. is added, whereby the mass is gradually converted into paste, which, after continued boiling and the further addition of lye of  $14^{\circ}$  B., becomes thicker and shows a clear uniform appearance. When a sample of the paste upon a glass plate remains quite clear up to the congealing point and causes a slight burning sensation upon the tongue, the saturation of the paste with alkali may be considered complete (about 2500 pounds of lye of  $14^{\circ}$  B. suffice for the complete saponification and saturation of 1000 pounds of oil). The paste is now either boiled until entirely free from froth and then carefully separated, so that no froth is formed, with common salt or salt water of  $24^{\circ}$  B., the result being a thick grain free from froth; or the paste is boiled not entirely free from froth but salted out when it spins threads, and after removing a portion of the sub-lye the grain is boiled clear like tallow soap.

For marbled soap the slightly colored paste is brought into the frame and the latter covered after thorough crutching. For smooth soap the grain is well ground by crutching in hot water, or, what is still better, potash lye of  $3^{\circ}$  to  $4^{\circ}$  B. until it has acquired sufficient fluidity to allow all the impurities to pass into the sub-lye.

Soap prepared from palm-oil soon develops an agreeable odor of violets and yields a thick, fat lather. 100 pounds of palm-oil yield on an average 150 to 155 pounds of soap, which is augmented about 10 pounds by grinding.

Besides bleached palm-oil the crude oil is also frequently used in England. Soap prepared from it retains the odor of palm-oil, and has the disagreeable property of causing brownish stains difficult to remove in clothes remaining long in contact with it.

In England the boiling of palm-oil is effected in steam-kettles with double sides, from six to eight of such kettles being connected with the steam-boiler. The large quantity of free fatty acids in palm-oil, which is still further augmented by bleaching, facilitating and promoting saponification, lyes of  $18^{\circ}$  to  $20^{\circ}$  B., which, however, should contain no free sodium carbonate, is therefore used from the start.

Fitting is effected by two methods: the paste is either salted out in the same manner as tallow-curd soap or Marseilles soap, or, as

is more frequently done, the soap is separated from the sub-lye by stronger lyes.

According to Stoeckhardt, 1, 2, and 4: Schaedler, 3, 5, and 6.	1	2	3	4	5	6
	PURE PALM-OIL SOAPS.					
	Curd-soaps.			Ground-soaps.		
	Bleached.	Un-bleached.	Un-bleached.	Bleached.	Marbled.	Un-bleached.
	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>
Water . . . . .	24.8	19.9	15.6	35.4	40.3	29.4
Fatty acids . . . .	61.2	65.2	72.5	49.9	45.8	55.0
Sodium oxide—						
Fixed . . . . .	8.0	8.6	8.6	7.0	6.5	6.5
Free . . . . .	1.7	1.2	0.2	1.0	—	1.2
Foreign salts . . .	1.3	1.8	3.1	1.1	0.9	4.5
Residue . . . . .	3.0	3.3	—	5.6	6.5	3.4
	100.00	100.00	100.00	100.00	100.00	100.00

Soap prepared from pure palm-oil being somewhat hard and brittle, the oil is generally worked in connection with softer fats, such as bone fat, olein, lard, peanut-oil, etc., or with 15 to 25 per cent. of resin. In the following we will describe a few such soaps prepared from palm-oil with an addition of other fats or resin.

*Stettin palm-oil household soap.*—Eight hundred pounds of palm-oil and 200 pounds of bone fat and 500 pounds of lye of 12° B. are brought into the kettle and combined by crutching over a slow fire. As soon as the mass is thoroughly combined more lye of 12° to 14° B. is added, and the whole allowed to boil thoroughly, the addition of lye, with vigorous boiling, being continued until a pale, clear paste is formed and a sample of it brought upon the tongue shows a slight touch. For the saponification of the 1000 pounds of fat about 2500 to 2600 pounds of lye of 12° to 14° B. suffice. Now add to the saturated, clear soap-paste, with constant vigorous stirring, in small portions, 150 pounds of pale comminuted resin and 150 pounds of caustic soda lye of 20° B., and allow the whole to combine with gentle boiling. When, after a few hours, the paste is clear and shows a



slight touch and a sample "wets" (shows moisture) and slides upon a glass plate, salt well out with salt water of  $24^{\circ}$  B. and boil the resulting grain clear. Now cover the kettle, and after allowing it to stand for a few hours to give the sub-lye time to settle, ladle the grain into small, low frames so that it cools rapidly. The soap is cut into rectangular pieces and pressed on all four sides.

Many manufacturers prepare this soap in the cold way. The process is briefly as follows: 50 pounds of bleached palm-oil and 7 pounds of pale resin are brought into the kettle and melted. After cooling to about  $111^{\circ}$  F., the fatty mass is combined by stirring with 10 pounds of caustic soda lye of  $38^{\circ}$  to  $40^{\circ}$  B. Now add 20 pounds of soda water-glass of  $38^{\circ}$  B., stir in 20 pounds of caustic soda lye of  $38^{\circ}$  to  $40^{\circ}$  B., and bring the well-combined soap-mass into the frame. If the greater portion of the lye were immediately combined with the fat and the water-glass then added with a few pounds of lye, the mass would become thick and difficult to bring into the frame, which is prevented by the simultaneous addition of lye and water-glass. This soap is also cut into rectangular pieces and pressed, and after lying for some time shows an agreeable violet odor. It is, of course, not a curd-soap.

*Soap from palm-oil and bone fat.*—Bring about 450 pounds of bleached palm-oil and 150 pounds of pale, purified bone fat into the kettle, and add about 350 pounds of lye of  $12^{\circ}$  to  $14^{\circ}$  B., and let all thoroughly combine over a moderate fire. Combination being effected, increase the fire and add more lye of  $14^{\circ}$  B., continuing this until a light, clear paste is gradually formed. When the paste is completely saturated with alkali, and a sample upon a glass plate remains clear until it congeals, and shows touch, boiling is continued for a little while longer, after which the paste is salted out. The soap being separated, a portion of the sub-lye is pumped out and the grain boiled clear in the same manner as tallow soap. Clear boiling being finished, the kettle is covered and allowed to stand a few hours to give the sub-lye time to settle.

The paste is then brought into low frames (only about four

inches high) and the surface of the cooled soap scarred with a round stick to give it the desired rough appearance.

*Soap from palm-oil and resin or colophony.*—This soap has a beautiful wax-like appearance, is readily soluble on account of the addition of resin, yields a stiff lather, and possesses good detergent properties. For the manufacture of the soap bring 425 pounds of bleached and 75 pounds of crude palm-oil and 250 pounds of tank-lye or caustic soda lye of  $14^{\circ}$  B. into the kettle and combine them over a moderate fire. Then add more lye of  $14^{\circ}$  B. and increase the fire. After continued boiling and adding more lye, the mass gradually forms into paste, which becomes thicker and clearer the further its saturation with alkali progresses. The oil being completely saponified, which requires about 1250 pounds of lye of  $14^{\circ}$  B., and the thick, clear paste showing a slight touch, 150 pounds of comminuted pale resin and caustic soda lye of  $20^{\circ}$  B. are added in portions until the paste is thoroughly fitted, a very moderate fire being kept up during the operation. For the saponification of the resin about 150 pounds of caustic soda lye of  $20^{\circ}$  B. will be required.

The sufficient fitting of the paste is recognized not only by a slight touch, but better and surer by a sample upon a glass plate, which, with thorough fitting, will slide upon the glass and show moisture.

The fitted paste is thoroughly salted out and the resulting grain, after pumping out a portion of the sub-lye, boiled clear so that it lies thick and free from froth in the kettle and a sample shows solidity and pressure.

For marbled soap or soap showing sheaves and flowers, the paste, after clear boiling and settling of the sub-lye, is ladled into the frames and the latter are covered. The manipulation of marbling, etc., is then commenced in the same manner as described for tallow soap.

For smooth soap the clear-boiled grain has to be ground. For this purpose the greater portion of the sub-lye is removed and the grain ground with hot water until the sub-lye begins to thicken and the grain is completely dissolved and lies bright in the kettle. A sample should show sufficiently solidity, but no touch. In grinding care must be had that the soap does not be-

come too pasty or too soft. After settling and resting for some time, the ground soap is ladled into the frames.

Some manufacturers prepare this soap by the following process: The palm-oil is first completely saponified with lye of  $14^{\circ}$  B. The clear fitted paste is then salted out, and, after removing the sub-lye, the resin is added and saponified with caustic soda lye of  $20^{\circ}$  B. The soap, after salting out, is boiled until it is free from froth and clear, and a sample is solid with a good pressure. The soap is finally ground with some water. This process also yields a beautiful product.

*Palmitin soap.*—Under this name a soap is brought into commerce by some manufacturers, which is prepared chiefly from palm-oil fatty acid in connection with a small quantity of other fats. Palm-oil fatty acid is a white solid fatty substance, and is obtained by withdrawing the glycerin from palm-oil, and subjecting the crude fatty acid obtained to distillation with superheated steam.

As fatty acids saponify very quickly, the manner of preparing soap from them varies somewhat from that employed for neutral fats, saponification being best effected by means of stronger lye containing more carbonic acid.

A suitable method of boiling is as follows: 2000 pounds of lye of  $18^{\circ}$  B. (required for 1000 pounds of fat consisting of 3 parts of palm-oil fatty acid and 1 part of saponified olein) are brought into the kettle and heated to boiling. The above quantity of fatty acids is then added in portions with constant crutching to the gently boiling lye and at once saponified. In adding the fatty acids the utmost care is necessary not to add a fresh portion before the preceding one has been completely absorbed by the lye. When a clear paste is formed, it is fitted to touch, allowed to boil for some time longer, and then salted out. The resulting grain is then boiled clear in the previously mentioned manner, and, after removing a portion of the sub-lye, ground with water until a cohesive liquid mass is formed. The soap is now allowed to rest for twenty-four hours in the covered boiler, and then carefully ladled from the thinly-fluid paste beneath it into large frames and covered. In cutting, the soap



shows a silvery radiated appearance and also an agreeable odor. The yield from 100 pounds of fat is about 160 pounds of soap.

*Resin curd-soaps.*—The resins used are galipot and colophony. They liquefy at the boiling heat of water, and saponify more readily and quicker than the fats. Saponification can be effected by means of sodium carbonate or caustic soda, the latter being, however, preferable.

The formula  $C_{40}H_{60}O_4 = 608$  molecular weight may stand for common resin or colophony, which is a mixture of several isomeric acids; 1 molecule of acid saturates 1 molecule of sodium or potassium oxide, therefore 608 parts, 62 parts of sodium oxide or 94 parts of potassium oxide, or 100 parts of resin require 10.2 parts of sodium oxide or 15.5 parts of potassium oxide for saponification, or 220 pounds of resin, 220 pounds of soda lye of  $19^\circ$  B., or the same quantity of potash lye of  $17^\circ$  B. The resulting product is not an actual soap, it having no consistency, and cannot be used without injury. With an excess of alkaline carbonates common salt separates upon the surface a soft thickly-slimy mass, while a portion of the soap remains dissolved in the sub-lye into which pass also in an unaltered state the brown coloring substances, as well as certain constituents of the resin. However, if resin is saponified in connection with other fats, common salt solution separates out all the resin-soap together with the fat-soap.

A solid soap cannot be prepared from pure resin, strong alkaline lye not separating pure resin-soap but remaining mixed with it, and hence the soap remains always smeary. It does not dry on exposure to the air, and the power of resin-soap artificially dried of absorbing water is so great that it deliquesces.

Though resin-soap by itself is of no value, an excellent and solid product is obtained by combining it in certain proportions with tallow-soap or palm-oil soap. The most suitable proportion of resin to fat is one-third; with equal parts the property of pure resin-soap predominates more or less, the soap becoming too soft.

The following may serve as combinations :—

	Parts.		Parts.
I. Palm-oil . . .	50	II. Palm-oil . . .	50
Tallow . . .	30	Cotton-seed oil . .	20
Resin . . .	20	Resin . . .	30
	Parts.		Parts.
III. Palm-kernel oil .	40	IV. Tallow . . .	25
Palm-oil . . .	20	Palm-oil . . .	25
Resin . . .	40	Resin . . .	50

In England the crude palm-oil is melted in kettles and pumped into cast-iron reservoirs, where it is kept liquid for the impurities to settle. The reservoirs are placed near the soap-kettle, and are provided with a float-gauge, which indicates how much oil is discharged. For  $3\frac{1}{2}$  parts of palm-oil 1 part of resin is used.

Resin-soap having a brownish-yellow color it is not necessary to bleach the palm-oil, and other waste fats, such as bone fat, wool fat, etc., can also be utilized. However, if bleached palm-oil is to be used it is best in order to obtain a white soap to first purify and bleach the resin.

Resin soaps are prepared according to different methods, a few of which will be given in the following:—

*Resin curd-soap* (30 pounds resin to 100 pounds fat).—An old method of preparing resin-soap, which was formerly much in use, is as follows: Combine by moderate boiling 1000 pounds of tallow and 150 pounds of crude palm-oil with 700 pounds of lye of  $10^{\circ}$  B. When combination is complete increase the fire and add gradually to the boiling mass lye of  $12^{\circ}$  to  $14^{\circ}$  B. until a clear paste is formed. When the paste is thoroughly saponified, continue boiling until it lies free from froth in the kettle, and a sample shows clear and solid upon a glass plate and causes a slight touch when brought upon the tongue. At this stage salt out the paste and the result will be a thick grain free from froth. Allow it to rest for some time in the covered kettle for the sub-lye to settle.

In the meanwhile saponify in another kettle 350 pounds of resin by adding lye of  $20^{\circ}$  B. (about 650 pounds) until the complete saponification of the resin is indicated by lye separating from a sample. As the separated lye absorbs all the impurities and the greater portion of the coloring substance of the resin, the resin-soap obtained is always of a light color.

The sub-lye is now pumped off from the grain-soap in the other kettle and the dirty lye from the resin-soap. Then add the resin-soap to the grain-soap, and mix both by vigorous stirring or the admission of steam. The soap is generally boiled upon a second water by liquefying the grain with lye of  $6^{\circ}$  to  $7^{\circ}$  B., then boiling for some time, and again concentrating it with lye of  $2^{\circ}$  to  $3^{\circ}$  B. so that a somewhat thin precipitate of paste is formed. After allowing the soap thus obtained to rest several hours it is framed; when cut it is of a beautiful appearance and great solidity.

*Resin curd-soap* (40 pounds resin to 100 pounds fat). A beautiful and cheap resin-soap is prepared by the following process: 500 pounds of bone-fat, 400 pounds of tallow, 100 pounds of crude palm-oil, and 700 pounds of lye of  $12^{\circ}$  B. are brought into the kettle and intimately combined by crutching over a slow fire. The fire is then increased and lye of  $14^{\circ}$  B. is gradually added until a clear, homogeneous paste is formed. This will require about 1800 pounds of lye of  $14^{\circ}$  B. The addition of lye is best executed by allowing one portion to thoroughly combine with the mass before adding another. When a sample of the paste remains clear for some time upon a glass plate and shows a slight touch, vigorous boiling is continued until the paste is quite free from froth. The paste is then salted out, about 8 to 10 per cent. of salt of the fat used being sufficient for the purpose. After thorough settling the sub-lye is pumped out or drawn off. The caustic soda lye of  $20^{\circ}$  B. (about 400 pounds) required for the 400 pounds of resin is then added and the soap boiled clear. The comminuted 400 pounds of resin are then added in portions and saponified with a moderate fire. The soap is then boiled until it lies free from froth in the kettle and shows good pressure. It is then thoroughly fitted and separated, and finally somewhat ground with water. The saponification of the resin with strong lye is of advantage, since the formation of froth is promoted by the great content of moisture brought into the soap with the use of weak lyes.

*Resin curd-soap* (50 pounds resin to 100 pounds fat).—Nine hundred pounds of tallow, 100 pounds of crude palm-oil, and 500 pounds of pale resin are brought into the kettle and com-



combined with 600 pounds of caustic soda lye of  $15^{\circ}$  B. with a moderate fire. Combination being established, increase the fire somewhat and add lye of  $15^{\circ}$  B. in portions until a quite clear paste is formed. Then add caustic soda lye of  $25^{\circ}$  B., and boil until the paste lies free from froth in the kettle, and a sample pressed in the palm of the hand leaves moisture behind, which is a better and surer test than the touch, of the complete saturation of the paste. After salting out the soap is boiled thick and clear, and the resulting grain is ground with some hot water. After standing about twelve hours in the covered kettle the soap is framed and crutched until cold.

*Resin curd-soap, according to the American method* (100 pounds resin to 100 pounds fat).—Nine hundred pounds of tallow and 100 pounds of crude palm-oil are brought into the kettle and intimately combined, by a moderate fire, with about 600 pounds of lye of  $12^{\circ}$  B. The fire is then increased and lye of  $12^{\circ}$  to  $13^{\circ}$  B. added in portions (each time about 200 to 300 pounds) until a good clear paste is formed. After withdrawing by continued boiling superfluous water from the paste, and the latter lies free from froth in the kettle and shows a slight touch, it is salted out. The sub-lye is then removed, and about 900 pounds of caustic soda lye of  $25^{\circ}$  B. are added to the grain, and then gradually the 1000 pounds of comminuted resin. The whole is then boiled until the resin is thoroughly saponified. By fitting to a good touch with lye of  $25^{\circ}$  B. a quite solid soap is obtained. The sub-lye is then again removed, the soap thoroughly ground with hot water, again fitted, and finally allowed to rest in the well-covered kettle for 24 to 36 hours. The soap is then brought into the frames, and having become somewhat soft by grinding a strong solution of crystallized soda (about 5 or 6 pounds to 100 pounds) is crutched in which renders the soap firmer. Sometimes 1 to 2 pounds of soda-water glass are added to the soda solution, which prevents the disintegration of the soap and is beneficial to its appearance.

*Dark resin curd-soap.*—For the preparation of dark resin-soap, which is frequently used for household purposes, scrubbing, etc., kitchen-fat, wool-fat, fuller's-fat, bone-fat, palm-kernel oil, and dark resin are generally employed.

The boiling of this soap is mostly executed by combining, for instance, 500 pounds of bone-fat, 200 pounds of kitchen-fat, and 300 pounds of wool-fat with about 500 pounds of lye of  $12^{\circ}$  B., and boiling the mass over a stronger fire, and with a further gradual addition of lye of  $12^{\circ}$  to  $15^{\circ}$  B., to a good paste. When saponification is complete and a sample shows a slight touch, 200 pounds of dark resin and about 180 pounds of caustic soda lye of  $20^{\circ}$  B. are added and allowed to combine over a moderate fire. The paste is allowed to boil vigorously for some time, and then fitted by carefully adding lye of  $25^{\circ}$  B. until a sample leaves moisture behind and slides upon a glass plate. The paste is then salted out, the resulting grain boiled clear in the usual manner, and the evaporated thick soap ground with hot water until the sub-lye commences to paste. For marbled soap the paste is brought into the frames, drawn through with an iron rod, and well covered.

Having given in the foregoing the various methods of boiling resin-soaps, it remains to say something in regard to the fabrication of these soaps. First, it is always advantageous to use only pure fat and resin. To attain this it is best to melt the fat upon water and dissolve the resin in the hot fat. After such an operation it will be generally found that the water is not only strongly colored, but that a thick sediment has formed upon the bottom of the kettle. Secondly, it is necessary in order to prevent the formation of froth to saponify the resin with strong caustic soda lyes, and besides care must be had to saturate the resin by a successive addition of lye. Thirdly, the fitting of resin-soaps must be only slight, since soaps with an excess of soda decompose. For a similar reason the use of caustic soda lye for resin-soaps is recommended, as this will frequently prevent sweating, for only soaps containing sodium chloride or much sodium carbonate, which attract moisture from the air, sweat.

In the following we give a few analyses of resin-soaps :—

According to Ure, 1; Stein, 2 and 3.	1	2	3
	RESIN-SOAPS.		
	Brown tallow and resin.	English.	Light palm-oil and resin.
	<i>per cent.</i>	<i>per cent.</i>	<i>per cent.</i>
Water . . . . .	23.50	22.23	26.94
Fatty acids, etc. . . . .	70.00	62.95	63.42
Sodium oxide . . . . .	6.50	8.03	7.19
Foreign salts and residue . . . . .	—	6.79	2.45
	100.00	100.00	100.00

*Olein-soap*.—For the manufacture of candles only solid fatty acids can be used, and, therefore, after the decomposition of the fats, oleic acid is obtained as a by-product by pressure. The oleic acid thus obtained is a mixture of a small quantity of solid fatty acids (palmitic and stearic acids) with a large quantity of actual oleic acid and its product of decomposition, sebacic acid, etc., and other organic substances formed by the action of sulphuric acid upon the fat. Commercial oleic acid, also erroneously called “olein” or “elain,” is of a brown color due to the absorption of oxygen from the air, and can be saponified with the carbonates of the alkalies as well as with caustic alkalies. The treatment with the carbonates is, however, more difficult, since strong foaming and consequent boiling over are caused by the escape of the carbonic acid.

Two varieties of oleic acid are found in commerce, saponified oleic acid and distilled oleic acid. The first variety, which generally contains a few per cents. of stearic acid, gives quite solid soaps with a yield of 150 pounds of soap from 100 pounds of fat, while the distilled oleic acid gives generally a soft product with a smaller yield (about 140 pounds). The latter is, therefore, principally used in connection with palm-kernel oil for smooth soaps, whereas good soaps can be prepared from saponified oleic acid without the addition of other fats.

The manner of preparing pure olein soap is similar to that given for olive-oil and palm-oil fatty acid. The most simple and advantageous method of manufacture is as follows:—



Tank-lye of  $22^{\circ}$  B., prepared with 50 to 52 per cent. of lime, is brought to from  $27^{\circ}$  to  $28^{\circ}$  B. by caustic soda. Nearly the entire quantity of lye required (about 1 pound lye to 1 pound oleic acid), with some salt, is brought into the kettle and the oleic acid added in portions, while the lye is kept in constant ebullition. By these means a uniform combination is effected, and by the use of strong lye the formation of froth prevented. If, after the complete saponification of the oleic acid and fitting to touch, some salt is wanting, it is added, and the soap will then be generally found lying in the kettle as a good grain free from froth, so that clear boiling can in most cases be omitted. After thorough settling the sub-lye is pumped out or drawn off and some lye of  $7^{\circ}$  B. is brought into the kettle, with which the grain is boiled for some time, so that it becomes completely saturated with alkali. The soap is then ground with water until the sub-lye commences to paste slightly. The kettle is then covered and allowed to stand 12 to 18 hours, when the soap is carefully ladled off from the slight precipitate into the frames and the latter are covered.

On cutting, the soap thus prepared shows a fine silvery radiated appearance and is delicate and quite firm; but if it is to be cut into square pieces and pressed or stamped, it has to be entirely smooth. For this purpose it is allowed to stand in the kettle for 24 to 30 hours, and is crutched in the frames until cold.

If, in order to increase the solidity of the soap, some bleached palm-oil is to be added, it can only be done after the oleic acid has exhausted the strength of the strong lye in the kettle, when the palm-oil is added together with the required lye.

Before subjecting the oleic acid to saponification it is frequently converted into the isomeric elaidic acid by means of nitrous acid, a small quantity of the latter sufficing for the conversion of a large quantity of oleic acid into the isomeric combination, which is as hard as tallow. It yields a beautiful soap which resembles tallow-soap and can be worked into grain-soap or ground soap.

*Soap from fuller's fat.*—A soap has been recently introduced into commerce which is prepared chiefly from fuller's fat, and, on account of its large content of fatty acid (about 62 per cent.), is

much used in the cloth industry and sometimes for household purposes.

Fuller's-fat is recovered by decomposing the soap-waters from cloth mills with sulphuric acid, a product of quite a light color being obtained after passing it through various operations. The value of fuller's fat varies very much, according to the quality of the soap used in the mills. The color of the fat is also of importance, as dark fat requires to be boiled upon several waters, and the appearance of the resulting soap is less desirable. Fuller's fat being a pure fatty acid, is readily saponified in a similar manner as oleic acid, and under certain conditions may yield a good grain-soap. Generally, however, it is boiled in connection with other solid fats, the resulting soap having a better appearance and feel.

Soap from fuller's fat is generally prepared by bringing about 1000 pounds of tank-lye of  $15^{\circ}$  B. into the kettle, and after heating to the boiling point, adding 50 to 60 pounds of common salt. The fire is then moderated and the fuller's-fat (about 500 pounds) gradually introduced. In adding the fat great care must be observed and water or weak lye always kept ready on hand, as on account of the escape of carbonic acid the mass may foam up and run over. Saponification is generally complete after crutching in all the fat. A small excess of lye is of advantage, as it promotes the decolorization of the fat.

The soap lying in the kettle as a small round grain is allowed to boil for some time with a moderate fire, when 50 pounds of comminuted resin and about 40 pounds of caustic lye of  $20^{\circ}$  B. are added. Boiling is now continued until a round grain is again formed, which is fitted. After extinguishing the fire and allowing the sub-lye to settle, the soap is ladled into large barrels and the dirty sub-lye allowed to run off. The soap, together with some other cheap fat and soap offal, is then returned to the clean kettle, sufficiently evaporated by gentle boiling, and carefully and slightly fitted with caustic lye of  $20^{\circ}$  B. When a sample, after cooling, is dry and without sharpness, the operation is finished and the soap brought into the frames.

For a lighter and more solid soap the resin is omitted, and after removing the sub-lye and thoroughly cleaning the kettle,

about 250 pounds of lye of  $25^{\circ}$  B. are added and the whole allowed to boil. Now add to the boiling grain, carefully and gradually, about 100 pounds of oleic acid, and when this is thoroughly dissolved, 150 pounds of palm-oil or palm-oil fatty acid. The soap, which will soon be thoroughly combined and liquid, is then salted out, boiled clear, and somewhat ground with weak lye.

Some factories purify the fuller's-fat by distillation with superheated steam and bring it into commerce under the name of "solid white olein." It is principally used for grain-soaps upon a precipitate of paste and resin grain-soaps, which are firm and of good appearance.

*Wool-fat soap.*—Under the name "wool-fat" a brown, sticky, and quite solid fat is brought into commerce, which is obtained by washing crude sheep's wool with soap and decomposing the resulting wash-water with acid, etc. Wool-fat is not counted among the actual fats, since, when boiled by itself, it does not yield serviceable soap. In connection with other fatty substances it yields, however, a fair curd-soap. To obtain wool-fat entirely free from water and dirt it is frequently purified by boiling it for some time upon salt water of  $16^{\circ}$  B. and carefully ladling it off the next day.

To manufacture curd-soap with a portion of wool-fat the following process is used: 250 pounds of bone-fat and 50 pounds of crude palm-oil are combined with 150 pounds of lye of  $12^{\circ}$  B. over a moderate fire. The fire is then increased and lye of  $12^{\circ}$  to  $15^{\circ}$  B. gradually added until a clear paste free from froth is formed, which is fitted to a slight touch. About 200 pounds of wool-fat and the required 200 pounds of lye of  $22^{\circ}$  B. are then added to the paste, and after thorough combination is established the whole is allowed to boil some time longer. Now slacken the fire and add gradually 50 pounds of comminuted resin, together with about 40 pounds of caustic soda lye of  $20^{\circ}$  B., carefully fit the paste after further boiling with caustic soda lye of  $25^{\circ}$  B. until a sample shows moisture, and then salt out. The dark sub-lye is now removed and a few bucketfuls (only sufficient to prevent scorching) of light sub-lye derived from curd-soap are thrown into the kettle, and boiling is continued



until a thick grain free from froth and without sharpness is formed. The soap is then brought into the frames, drawn through with a rod, and well covered.

Wool-fat is also purified in the same manner as fuller's-fat by distillation, and then comes into commerce under the name of "cholestrin." It is a solid, white product, and on account of its better appearance can be used for various light hard soaps.

*Curd-soap from fish-tallow.*—Under the names of "whale-fat" and "fish-tallow," two fatty substances have been recently brought into the market. Several soap-boilers have made experiments with these fats, but on account of their intense fishy odor, which strongly adheres to soaps manufactured from them and the present low price of tallow, their general introduction is not likely.

Of the two fats fish-tallow, which is the stearic portion obtained by pressure from fish-oil, deserves the preference, since, in connection with other fats and resin, it may serve for the preparation of yellow soaps of fair quality. It is of a brownish color and quite firm, and saponifies with tank-lye as well as with caustic soda-lye. 100 pounds of fish-tallow yield about 130 pounds of curd-soap.

The manufacture of yellow curd-soap is generally effected in the following manner: About 400 pounds of fish-tallow, 25 pounds of crude palm-oil, and 250 pounds of lye of  $12^{\circ}$  B. are brought into the kettle and combined over a moderate fire. The mass is then allowed to boil vigorously, more lye of  $15^{\circ}$  B. being gradually added until paste is formed. The paste is vigorously boiled, with the addition of the lye still required, until it is clear and free from froth, and is then fitted to a slight touch with caustic soda lye of  $20^{\circ}$  B. The fire is then moderated, and 50 pounds of comminuted resin are gradually added, together with 40 pounds of caustic soda lye of  $20^{\circ}$  B. When the resin has combined with the rest, boiling is continued with a further addition of lye of  $20^{\circ}$  B. until a sample of the paste shows moisture, which indicates the complete saponification of the resin. Salting out is then proceeded with. For this purpose salt soaked in water is gradually added until separation is effected and the grain boils in beautiful laminae, and a sample pressed in the palm

of the hand does not smear but is firm and dry. After allowing the soap to rest for some time, the very dark and jelly-like sub-lye is removed and a few bucketfuls of light sub-lye derived from a white curd-soap are brought into the kettle. Boiling is now continued until a thick grain free from froth and without sharpness is formed, which, after settling, is ladled into the frames and well covered. By clear boiling upon salt-lye the grain acquires a considerably lighter color, and by the addition of resin the odor of train-oil becomes less perceptible. The odor of fish-tallow is, however, so powerful as to make it impossible to use large quantities of it, as even with an addition of only ten per cent. the odor of train-oil is clearly perceptible.

*Apollo soap.*—To prepare the so-called Apollo soap, which is a ground olein-soap, the quantity of lye of  $20^{\circ}$  to  $22^{\circ}$  B. required for the saponification of the oleic acid to be used is brought into the kettle and some salt added. The object of the addition of the latter is to prevent a complete pasting of the mass, since this would be a hindrance to the complete saponification of any oleic acid which may still be wanting.

The process of fabrication is briefly as follows: 5000 to 5300 pounds of lye of  $22^{\circ}$  B. are brought into the kettle and heated to boiling. Five thousand pounds of oleic acid are then gradually added with constant crutching. Saponification takes place almost instantly and without the formation of froth. The soap boils either in dark broad laminæ, or, if the fire is somewhat strong, in grains. At this stage a few bucketfuls of lye of  $5^{\circ}$  to  $6^{\circ}$  B. are added, which makes the saponification more complete. The soap is then fitted to a slight touch and salted out. It now lies as a beautiful, thick grain of a honey-yellow color in the kettle and might be considered finished, but is generally allowed to rest three to four hours in the kettle for the sub-lye and the dirt to settle.

After removing the sub-lye and the dirt about 2000 pounds of soda lye of  $5^{\circ}$  to  $6^{\circ}$  B. and sufficient salt are brought in the kettle, so that the soap remains separated and will not form froth. It is then again thoroughly boiled. The soap is then fitted to a slight touch with lye of  $20^{\circ}$  B., and should it seem too grainy is

ground from above with some hot water until the grain is liquefied.

The soap is now more solid and shows a good pressure. It is then crutched through and allowed to stand, if possible overnight, in the covered kettle. The next day any froth thrown out is carefully removed and the soap brought into the frames.

In many factories small iron frames holding ten to twelve hundredweight are used and the soap is crutched until cold, whereby it is rendered entirely smooth and ready for cutting in three to four days, but shows no grainy silver stripes. Slow cooling in large frames well covered is to be preferred, though it is advisable to crutch the soap for a few minutes after it is brought into the frames. When the soap is cold it is cut into slabs, which are somewhat dried and then cut into bars. The latter are stamped, dried in the air, and stored for at least three to four weeks before they are brought into commerce. This soap is not only an excellent one for fulling cloth and for other textile purposes, but also for household use. It is largely manufactured in Austria, having been first introduced at the celebrated Apollo candle factory at Penzing, near Vienna, whence its name is derived. The material used there is the oleic acid obtained as a by-product in the manufacture of stearin, and is brought into commerce under the name of "Vienna eläin." It is of a pale yellow color, very rich in stearin, and yields excellent soaps.

*Turpentine soap.*—This soap was formerly prepared from equal parts of tallow and palm-oil, 8 to 10 per cent. of cocoanut-oil, and 20 to 25 per cent. of pale resin. The tallow and palm-oil were saponified with lye of 15° B., boiled clear, and the resin soap, previously prepared in another kettle, crutched in. The whole was then strongly fitted and the soap ground with 8 to 10 per cent. of cocoanut-oil. The resulting soap was very good, but would be too expensive at present prices.

Since the introduction of the cheap palm-kernel oil and caustic soda the following process is generally adopted :—

Two thousand pounds of palm-kernel oil and 600 pounds of pale resin, previously comminuted, are saponified with about 4000 pounds of caustic soda lye of 23° B. The resin combining very



quickly and readily with the strong lye, facilitates the combination of the palm-kernel oil with the alkali.

The soap is now fitted to a slight touch. About 80 to 100 pounds of crude palm-oil, together with the required lye, are then added for coloring, and after boiling until the froth entirely disappears, the soap is strongly salted out with caustic soda lye of  $35^{\circ}$  to  $36^{\circ}$  B. and some salt. The resulting stiff grain is then ground with hot water. The kettle is then covered and the soap allowed to stand over night for the sub-lye to settle. The next day the soap is framed and crutched for about one hour. Should any sub-lye be inadvertently ladled into the frame, add a few pounds of palm-kernel oil and crutch thoroughly. Before allowing the soap to rest it is perfumed with five pounds of oil of turpentine and then well covered.

*Russian saddle soap.*—This soap is a specialty with some German manufacturers. The following materials are used: 1000 pounds of Russian tallow, 400 pounds of crude palm-oil, 560 pounds of Ceylon cocoanut-oil, and 400 pounds of pale resin. The soap is boiled either by the direct or indirect method.

For the direct method a sufficient quantity of caustic soda lye of  $25^{\circ}$  to  $26^{\circ}$  B. for the saponification of the above-mentioned materials is brought into the kettle. If any soap-cuttings are on hand they are allowed to dissolve in the lye. The tallow is then added; next the palm-oil, cocoanut-oil, and finally the resin.

Should the resulting paste be viscid and thick, add gradually lye of  $25^{\circ}$  to  $26^{\circ}$  B. until the paste is clear and well fitted. The soap is then boiled until laminae are formed. It is then salted out and ground with hot water until a sample "wets," *i. e.*, leaves moisture behind. The soap is now left in the well-covered kettle till the next day, when it is brought into small frames holding about three to six hundredweight each and crutched cold. It is cut into five-pound bars and stamped.

For boiling the soap by the indirect method, the tallow, palm-oil, and resin are brought into the kettle and saponified with soda lye of  $20^{\circ}$  B. The mass is then boiled to a clear paste which is fitted to a slight touch and salted out. The soap-cuttings are then dissolved in another kettle in lye of  $20^{\circ}$  B. and the cocoanut-oil added. The grain in the other kettle is then brought into the

one containing the soap-cuttings and cocoanut-oil and the soap is finished in the above-described manner.

*Sinclair's soap*.—Under the name of "*Sinclair's English cold-water soap*" an article has been recently introduced which, according to accompanying directions, offers great advantages for laundry purposes, since it renders the use of soda and boiling of the clothes unnecessary, rubbing being required only for very dirty articles. The soap, it is claimed, saves fuel and washing material and the work can be done in one-third the time. The soap is dark yellow and has a slight odor of artificial oil of bitter almonds.

Many contradictory analyses of this soap have been published, which may be partly due to the fact that such soaps are never manufactured alike even with the use of the same fats. One analysis shows the following constituents :—

Alkaline sebate (actual soap)	. . . . .	62.7 per cent.
Sodium carbonate . . . . .	. . . . .	1.3 "
Matter insoluble in alcohol . . . . .	. . . . .	2.6 "
Water . . . . .	. . . . .	32.4 "

According to this, the constitution of this soap does not differ from that of ordinary soaps and its use offers not the slightest advantage, the wonderful properties claimed for it being fictitious.

The soap is prepared by boiling 100 parts of fat (generally palm-oil) and 60 parts of resin to a stiff grain, and bringing the latter into frames of 12 to 13 cwt. capacity, where it is crutched until cold. Only when the grain is too stiff it is ground with solution of crystallized soda and a few per cent. of water-glass to render it more pliant. Imitations of Sinclair soaps are nearly all prepared by melting 100 parts of fat with 80 to 100 parts of resin and saponifying with caustic lye of 36° B. These soaps, however, are heavier and seldom successful, since the resin which remains unsaponified and sticky is perceptible in washing.

*Augmentation of curd-soaps*.—An increase of the yield (about ten per cent.) of curd-soaps is obtained by grinding, the soap absorbing water during this operation. The content of water incorporated by grinding should, however, not exceed a certain limit, as otherwise the soap becomes too pasty and soft, or, as it is technically termed, "over-ground."

In many factories the yield of curd-soaps and resin curd-soaps boiled clear upon sub-lye is artificially increased by crutching in substitutes, the operation being technically termed "*filling*." By this manipulation the soaps acquire a "dead" appearance, though their solidity and washing power are not injured to any great extent.

For such an artificial increase of curd-soap, materials must, of course, be chosen which possess the property of not injuring the appearance of the soap too much and of not drying too quickly. As especially suitable filling materials, water-glass, soda, and talc are recommended and generally used either by themselves, or, better, in connection with each other. Water-glass by itself has the property of giving a good appearance to fresh soap and protecting it from drying out too quickly, but by age the soap acquires a mean appearance and becomes hard as stone. An excellent filling material suitable for most curd-soaps is prepared by stirring 100 pounds of talc into 150 pounds of boiling water, then adding 30 pounds of crystallized soda, and gradually crutching in 140 pounds of soda-water glass.

The curd-soaps to be increased are best prepared from fats rich in stearin, such as tallow, palm-oil, fatty acids, etc. They are formed into a paste with the required quantity of lye, as previously described, salted out, and evaporated to a stiff grain free from froth, which is thoroughly ground with water. Thorough saponification of the fats used is absolutely necessary, since, if the soap shows the slightest defect in this respect, it can be filled only slightly or not at all. When the grain is sufficiently ground and dissolved and shows good pressure while the sub-lye remains clear, the kettle is covered and allowed to stand for some time for the contents to settle. After the removal of the sub-lye and the soap being cooled to about  $167^{\circ}$  to  $178^{\circ}$  F., 30 to 40 pounds of the above filling material are crutched in for every 100 pounds of fat used. As the soap to be filled does not turn out always alike and therefore absorbs more or less filling material, it is advisable, in order not to expose an entire boiling to danger, first to test the absorbing capacity of the soap by a small sample.

Good results in filling can only be obtained by great care and experience. Many soaps can be more readily and better filled by



using the filling material in a warm state, while others better absorb cold filling. The operation itself can be executed in the kettle after the removal of the sub-lye or in a suitable vessel.

In the same manner as tallow and palm-oil curd-soaps, resin curd-soaps prepared from the same materials with 30 per cent. of resin can be filled to the extent of about 30 per cent.

The filled soaps are brought into smaller frames than those employed for curd-soap, and, after cooling, cut and dried for some time, whereby they gain in solidity. They are then cut into square pieces, pressed, and brought into commerce.

### *b. Curd-Soaps upon a Precipitate of Paste.*

The fabrication of smooth curd-soaps, which are found so much in commerce at the present time, is effected by two different methods, according to the fats used. The object of both methods is to produce as pure a soap as possible by separating all the impurities with the superfluous content of water and alkali.

One method is to grind by means of water curd-soaps boiled upon sub-lye until a paste is formed, and is used for all soaps from animal fats, olive-oil, palm-oil, and oleic acid. The other method is based upon the formation of a precipitate of paste, which is produced by adding either lye or salt in excess, though in both cases not sufficient to effect a separation of the soap. This method is only applicable when working cocoanut-oil or palm-kernel oil in connection with other fats, and can be executed either in a direct or indirect way.

As in the fabrication of other soaps, the soap-boiler must here be guided by the nature of the fats to be used and the arrangement of the factory. What he has to attain is *a pure soap free from froth and stains and thoroughly settled with as little precipitate of paste as possible*, and, further, an utmost yield of good salable soap from the fats used. This is not an easy task, and requires quick observation and a thorough knowledge of the nature of fats. With the frequent fluctuations in the fat and oil trade it is no longer possible to work according to one established routine, and the production of a uniformly beautiful product answering all the above demands is difficult.

The large number of smooth curd-soaps brought into commerce renders it impossible to treat of them collectively. Special attention has to be paid to all possible contingencies, and hence the fabrication of smooth soaps from various combinations of fat must be described in such a manner as to render this volume useful for other times than the present, and serve, especially the younger soap-boiler, as a guide in carrying on his business.

All wax-soaps should be free from dirt and be lustrous, and hence fats free from impurities or previously clarified are chosen for their fabrication. Sometimes even this is not sufficient, and defects have to be remedied by the manner of boiling, which is frequently effected by first boiling the lardaceous or all the added fats to grain. Direct boiling is, however, the method most generally used, it having the advantage of being quicker, though it is doubtful whether it is always cheaper.

By direct boiling the entire operation is finished in a few hours, and as but very little lye is separated, only a small quantity of common salt is required. Furthermore, the consumption of fuel is small, as in working with strong lyes the heat developed during the combination of the fats with the alkalies is frequently so great as to render after-firing unnecessary. These points are in favor of direct boiling. If, however, on the other hand, the added fats are first boiled to grain, the boiling process, even with the use of two kettles, lasts much longer, and more lye, more salt, and more fuel are used; but the fact that the consumption of more lye requires also more alkali would prove that in many cases better results and a larger yield are obtained than by direct boiling.

Every soap-boiler knows that the yield depends less on the fats used than on the lyes. By boiling with caustic lye of from  $28^{\circ}$  to  $30^{\circ}$  B. it can be shown by calculation that for the saponification of 100 pounds of fat about 17 pounds of caustic soda of 66 per cent. are required, but with lyes of  $14^{\circ}$  to  $15^{\circ}$  B. at least 20 pounds. Even if a portion of the caustic soda passes into the sub-lye, the whole of the excess is not lost, but lies in the increased soap. While with strong lye a yield of 150 per cent. is scarcely obtained from the best fats, with weak lye a yield of 160 to 165 per cent. is of ordinary occurrence. Hence soaps boiled upon a precipitate of paste are the more firm and dry the

stronger the lye used for boiling, while most soaps boiled by the indirect method only acquire such firmness by after-drying. Though what has been said does not prove the absolute necessity of indirect boiling, it is indispensable for all fats requiring weak lye for their correct saponification, the principal ones of these being all those of animal origin. Oleic acid should also be first boiled to grain, since good olein soaps from oleic acid alone can only be prepared by repeated and long-continued boiling.

Direct boiling is best when much cocoanut or palm-kernel oil is used in connection with such light oils as cotton-seed or linseed-oil, because the process of boiling proceeds with greater regularity and the resulting product shows greater solidity. Indirect boiling becomes, however, absolutely necessary when the quantity of supplementary fats used is larger than that of the combining fats, and also when they consist of mixed fats, as, for instance :—

Tallow	.	.	.	.	.	.	.	400 pounds.
Cotton-seed oil	.	.	.	.	.	.	.	300 “
Peanut-oil	.	.	.	.	.	.	.	300 “
Palm-kernel or cocoanut-oil	.	.	.	.	.	.	.	500 “

Such a formula can, of course, be directly boiled, but while the resulting soap will show a firm cut surface and have a bright and clear appearance while warm, it will not improve by storing, but become dull and finally entirely lose the silvery flux. On the other hand, by indirect-treatment the resulting soap will be moderately firm, of a better appearance, and improve by age.

Supplementary fats (tallow, cotton-seed oil, etc.) being cheaper at the present time than cocoanut-oil or palm-kernel oil, such a combination as the above is of ordinary occurrence. The softer fats are also frequently replaced by lard, horse-fat, and bone-fat, and even by oleic acid or bleached linseed-oil, the use of the latter being by no means objectionable, since the resulting soap is soft to the touch, yields a strong lather, and possesses good detergent properties. A too large addition of it must, however, be avoided, as otherwise the soaps acquire a bad odor.

Indirect boiling being the most advantageous with the above formula, the process is as follows :—

The fats are boiled either with tank-lye of  $12^{\circ}$  to  $14^{\circ}$  B., or with caustic soda lye of  $10^{\circ}$  to  $13^{\circ}$  B., 220 to 250 pounds of



such lye being required for the complete saponification of 100 pounds of fat and for obtaining the utmost yield.

The tallow and other fats, except the palm-kernel or cocoanut-oil, having been brought into the kettle, one-eighth of the quantity of lye required is added and a moderate fire started. If combination does not take place immediately after ebullition, stronger boiling is of no use. The defect is remedied by adding some cuttings of the same kind of soap, or waiting with the furnace door open until combination takes place. Lye is then gradually added until a clear paste free from froth is formed. The addition of the correct proportion of lye is recognized by a grain-like separation of the soap. Water is then added until combination and paste become again apparent. If the soap is, however, still viscid and shows no taste of lye, lye is wanting. If one is sure of having added the required quantity of lye, some stronger lye, of about  $15^{\circ}$  B., is added and boiling continued until the clear soap breaks into regular roses.\* Salting out is now proceeded with, 5 to 6 per cent. of common salt being sufficient for the purpose. It is recommended to soak the salt in water and add it, with the furnace door open or steam shut off, by scattering it successively over the soap. Generally the grain rises up at once and the whole is then boiled once more until laminae are formed and the lye runs off clear.

With the use of good, pure fats the lye settles completely in two hours, when it is removed and the necessary quantity of lye for boiling the cocoanut and palm-kernel oils is brought into the kettle, about 600 pounds of caustic lye of  $20^{\circ}$  B. being required for the above quantity of 500 pounds of oil. After boiling the grain in the kettle with this lye, some soap-cuttings are added, and, when all is dissolved, the oil (400 pounds of palm-kernel oil and 100 pounds of cocoanut-oil can be recommended) is gradually introduced. If the soap, after the addition of the last portion of cocoanut-oil, should be viscid or glassy, strong salt water is carefully added until the soap breaks uniformly into roses. This addition of salt water is, however, not necessary if, as is fre-

\* The thickening soap on the place where the steam from below forces its way through resembles an expanding rose.

quently the case, the grain holds salt lye fixed. Such finished ground-soap should not show any touch of sharpness and must be evaporated until it begins to puff like soap containing little water. Salt soaked in water is then gradually added, 6 to 8 per cent. of the palm-kernel and cocoanut-oils used being frequently sufficient. The soap now becomes thinner and feels moist to the touch. If it yields any lye, separation has been carried too far, but this defect can be remedied by supplementary grinding with water. It is a good sign when, after the soap ceases to boil and remains covered for a few minutes, a bright grain lies in the kettle. The appearance of white or dull soap when scraping the surface with a crutch indicates that separation has been carried too far; it is remedied with water. Separation not carried far enough is, as a rule, not a defect. If in ladling out it is observed that there is too much paste in the kettle, some strong salt water may be added by allowing it to run down the sides of the kettle, and the grain separated thereby is mixed with the soap in the frames.

We would here remark that the quantity of lye prescribed (600 pounds of 20° B.) is, as a rule, too small for the saponification of 500 pounds of palm-kernel and cocoanut-oil, and, hence, it may happen that it is not enough or just sufficient, this depending entirely on the condition of the grain. If, on the one hand, the grain is oversaturated with strengthening lye, it will subsequently yield lye, and if, on the other, there is too little lye or just sufficient, the after-effect is shown in boiling for combination. Now as lye can be readily added, but a preponderance of it only be reduced by increasing the quantity of fat, it is better to prescribe too little than too much for these soaps.

The same holds good as regards the quantity of lye prescribed for the preparatory boiling to grain. The statements of areometers differ, and hence it may happen that though caustic soda lye of 10° to 13° B. is used, 280 to 300 pounds of it might nevertheless be required.

*Prime white curd-soap (wax-soap).*—This is one of the principal soaps which is best boiled upon a precipitate of paste. It can also be prepared by incompletely salting out a paste boiled largely from palm-kernel oil or cocoanut-oil, but the result is

generally not satisfactory, especially when using animal fats in connection with them, and a uniformly good product can only be obtained by a correspondingly correct manner of boiling. As a fine quality of soap can, however, be manufactured by the last-mentioned method from palm-kernel oil and oleic acid, we give the process, which is as follows:—

Lye of  $20^{\circ}$  to  $23^{\circ}$  B. prepared by treating 95 per cent. soda with 50 per cent. of lime, and a combination of one-third to two-fifths of pale oleic acid and two-thirds to three-fifths of palm-kernel oil are used. As tank-lye of the above strength contains much carbonate, it is best first to paste the palm-kernel oil to a strong touch and add at once some salt to prevent thickening. The oleic acid is then gradually brought into the kettle, a moderate fire being kept up, and after again fitting the paste to a perceptible touch, it is allowed to boil until the froth disappears, which will require only a short time. The paste which is perfectly clear, and boils black and bright without rising much, is thoroughly saturated, the fats being completely saponified and the alkaline carbonate of the lye having also entered the combination. Boiling is then finished by gradually adding with constant stirring some moistened salt until the paste “wets” perceptibly without lye draining off. In case the paste has been salted out too strongly, it is ground with some water, and then, after removing the fire, is allowed to stand a few hours in the well-covered kettle. When the lid is removed the soap in the kettle will be found to be of a honey-yellow color, free from froth and liquid, and ready for framing. With correct salting out a thin layer of black, liquid paste, which contains but little soap, is found between the soap and sub-lye, which is boiled and salted out with the sub-lye. If, however, a stronger precipitate of paste is found, some grain can be obtained by careful after-salting, which is crutched into the soap in the frames.

With some experience uniformly good results can be attained in this simple manner without much residue of grain-paste. It is not difficult to obtain nearly the entire yield, about 140 per cent., from the fats in salable, pure white soap, as the thin paste floating upon the lye contains, with a correct execution of the work, only the marble-forming dirt. This process is, however,



only available for the above combination, the result being not nearly so uniform by replacing the oleic acid by cotton-seed oil, tallow, or lardaceous fats.

By the direct method smooth white curd-soap is most readily prepared from palm-kernel oil. As soap from the pure oil is, however, very brittle and lean, it is best to use a combination of two-thirds to three-fourths palm-kernel oil and one-third to one-fourth of soft or liquid fats in order to make the soap more delicate and pliant, cotton-seed oil, oleic acid, lard, and peanut-oil being most suitable for the purpose. The following may serve for a formula: Palm-kernel oil 1000 pounds, cotton-seed oil 250, lardaceous fats 250.

For pure white soap the cotton-seed oil requires bleaching. For this purpose it is heated to 113° F. Dissolve for every 100 pounds of oil  $\frac{1}{2}$  pound of potassium bichromate in  $1\frac{1}{4}$  pounds of boiling water, add to the solution  $1\frac{3}{4}$  to 2 pounds of hydrochloric acid, and pour the mixture with vigorous crutching into the oil to be bleached. After remaining in contact with the bleaching liquid for about a quarter of an hour the mass assumes a bluish-green color. When a sample is dropped upon a glass plate, a dark-green precipitate is at once recognized, from which, after standing a short time, the oil runs off entirely colorless. After standing for one hour the clear oil is drawn off and at once used for saponification.

The lye for boiling smooth white curd-soap by the direct method is best prepared from pure, high-graded caustic soda, or by increasing the strength of good tank-lye of 15° to 18° B. with caustic soda to 30° B. As lye of this strength requires great care in effecting the combination, it is best first to bring the entire quantity of palm-kernel oil into the kettle, and after introducing combination with one-half the quantity of lye required (about 500 pounds) gradually add the remainder until saponification to a good touch is effected and the soap again boils thin. By now adding with a moderate fire the cotton-seed oil and other fats, which combine readily on account of the saponification already introduced, a thick and curly-boiling soap is soon formed, which is reduced to normal boiling by fitting with strong lye or lye containing salt. The soap is then fitted to a strong touch

until it boils perceptibly thinner, when it is somewhat evaporated and then shortened with moistened salt until, when thrown with the spatula, it flutters and bubbles. It is finished when a sample cooled upon the spatula feels moist. Soap prepared with lye of such strength and correctly treated will never show froth, the appearance of the latter being generally due to the use of too weak lye. In shortening the salt must be added carefully, observing the effect produced by one portion before adding the next. Soap too strongly separated frequently shows gray stains.

As regards the framing of this soap, it is best to ladle out small boilings after remaining at rest for one or two hours, in order to give the paste time to subside somewhat, and keep the soap in the well-covered frame for 24 hours. Boilings of 6000 pounds and over may be left in the kettle over night, any froth or cold foliated coating being removed before ladling out to prevent any cold soap from reaching the frame.

If the fats used are melted by steam or otherwise thoroughly purified, it is not necessary to pour the soap through a sieve; but where there is any danger of injuring the quality by impurities, bark, or wood from the barrels, it is best to use this precaution.

Soap thus prepared lies dark in the frame, but is clear as honey, and after remaining covered for 24 hours, and when sufficiently cooled off, is generally pure to the bottom and ready to be brought into commerce. Soaps prepared according to this process are sufficiently firm and at the same time delicate and pliant, and by storing acquire a beautiful white color. The yield is about 120 pounds of prime curd-soap from 100 pounds of fat used. The paste is best boiled out with pale oleic acid, and the resulting grain used for the same kind of soap or for a second quality. In summer it is advisable to use less cotton-seed oil and more palm-kernel oil.

The process of boiling with direct steam differing somewhat from the preceding will be described separately.

*Boiling smooth white curd-soap with the use of direct steam.*—Animal tallow always has been and is still at the present the best material for household soaps, and though pure tallow curd-soaps are now but little used for laundry purposes, tallow is nevertheless the material which gives to all soaps a certain solidity and

high economical value. For this reason it is the more valuable for smooth white soaps, as the product obtained is, independent of its white color, of excellent quality and yields the largest return. The materials originally used by Groodhaus, of Darmstadt, were tallow and cocoanut-oil, for which, however, with increasing competition, all possible fats and oils were substituted, so that in 1882 and 1883, when good animal fats were very scarce and dear, even linseed-oil was used. By the extension of the manufacture of half-boiled soap from 1860 to 1870, the spoiling of many boilings gave to the soap-boiler useful hints for the manufacture of smooth white soaps, so that the general introduction of these soaps into commerce may be dated from that time.

The production of a beautiful white soap is much facilitated by the use of steam, but as fluctuations in the price of fats and oils will frequently necessitate a change in the combination of the fats used, suitable measures as regards the employment of steam have to be taken.

We give in the following a few formulas which, on account of their composition, are likely to give uniformly good products.

	Parts.	'Parts.	Parts.	Parts.
Tallow free from dirt . . . .	10	10	10	10
Palm-kernel oil . . . . .	6-7	10	15	20
Cotton-seed oil . . . . .	1	2	3	5

The more cotton-seed oil is used the greater the necessity of its being first bleached. Bleached linseed-oil, white oleic acid, or double the quantity of lardaceous fats may be substituted for cotton-seed oil.

We would here remark that in 1877 and 1878, when American lard was very cheap in Europe, large lots of it were frequently used, especially for white curd-soaps, and it can be confidently asserted that the product brought into the market during this time was the finest ever seen.

Should the kettle not be large enough to work the entire quantity of fat at one time, it is necessary first to saponify the tallow and cotton-seed oil, for which lye of less strength may be used. With a large percentage of palm-kernel oil it is, however, better



to bring it also into the kettle and use stronger lye. For instance, 2000 pounds of tallow, 1000 pounds of palm-kernel oil, and 300 to 500 pounds of cotton-seed oil are pasted, in order to prevent in salting out any strength from passing into the sub-lye, either so as to be perfectly neutral or show only a moderate touch. By the admission of steam through a perforated pipe on the bottom of the kettle, and simultaneous use of an open fire, saponification takes place very rapidly. In order to prevent thickening it is advisable to compound the lye, especially when strengthened with caustic soda, with some salt, or scatter the latter, after combination is complete, over the surface of the contents of the kettle, this being absolutely necessary with the use of much palm-kernel oil. Salting out is commenced as soon as the paste is clear and shows no perceptible touch, and is continued until the lye runs off clear. Special measures for safety are not required with the use of much palm-kernel oil, and besides, boiling over can be readily prevented by shutting off the steam. The paste separates without perceptible heating in the grain or sub-lye, and after boiling for some time the steam is shut off and the fire removed and the kettle allowed to remain at rest for a few hours. The open fire is necessary so that in salting out none of the salt will remain ineffective and undissolved on the bottom of the kettle.

After removing the sub-lye and combining the remaining 2000 pounds of palm-kernel oil with about 1000 pounds of lye of  $26^{\circ}$  B., another 1000 pounds of the same lye are gradually added until a slight touch is perceptible, when the paste is then again salted out.

Clear boiling of the paste upon the sub-lye in the kettle is then commenced, which, however, can only be done over an open fire, since the condensed water of the steam would render the process more difficult. In doing this care should be had that the lye retains some strength. The less sub-lye there is, and the stronger it is, the quicker the froth is thrown off. The soap must not, however, to prevent scorching, be boiled dry. When the grain is free from froth and large-grained, soap-cuttings are added, which are allowed to dissolve, the kettle being covered in the mean while. The fire is then withdrawn, and after allowing the soap

to remain at rest for a short time, the under-lye is again drawn off or pumped out and any light froth upon the grain removed.

The operation of grinding is now commenced, and is carried on with high-pressure steam of about three and a half to four atmospheres. As soon as the steam is admitted hot water is quickly poured over the soap while a workman promotes the operation by vigorous crutching. When the mass begins to form a uniform thin paste, which runs from the spatula without showing dry spots, and which in throwing flutters and bubbles, the soap is finished. After boiling the soap through once more, the steam is shut off and the well-covered kettle allowed to stand quietly for 24 hours. In bringing the soap into the frames the same rules as previously given for soap boiled by the direct method are observed. The better the operation of grinding has been executed the more delicate, whiter, and more homogeneous the grain will be, but also the smaller the yield of *prime* soap. The grain resulting from salting out the paste remaining in the kettles is used with the next boiling.

*Second quality of smooth white curd-soap.*—Frequently a second quality of smooth white curd-soap is boiled from the precipitate of paste from the prime quality, which is best effected by boiling the precipitate with cotton-seed oil or pale oleic acid, allowing about 100 parts of oil to 300 parts of fatty contents of the precipitate. The paste formed is fitted to a slight caustic taste and then slightly salted out so that a complete separation is not effected. After allowing the soap to rest for a short time, it is ladled into the frames while hot. The small residue of precipitate is used for resin-soaps. The soap thus obtained is somewhat darker and generally less pure than the prime quality.

*Smooth curd-soaps with resin.*—For several years a large demand has sprung up for smooth curd-soaps with resin, so that in some regions they have superseded almost all other kinds of soap for household purposes. The manufacture of this soap has been materially facilitated by the introduction of high-grade caustic soda, which permits the production of a lye excellently adapted for the most rational process of boiling by strengthening tank-lyes. Though the combination of fats used, which consists chiefly of palm-kernel oil, requires strong lyes, this is still more

the case for the saponification of the resin. The non-observation of this leads readily to such frothy and badly-settled soaps as are only too frequently found in the market.

The best soaps of this kind are those which were formerly prepared by the direct method from pure palm-kernel oil, with a small addition of cocoanut-oil and 10 to 15 per cent. of pale, transparent resin, combined with caustic soda lye of  $27^{\circ}$  to  $28^{\circ}$  B. To 1000 pounds of palm-kernel and cocoanut-oil and 150 pounds of pale, transparent resin, were added 700 pounds of caustic soda lye of  $27^{\circ}$  to  $28^{\circ}$  B. After dissolving pure cuttings of the same soap in the mass, 1000 pounds of oil were gradually added. On account of the strong lye a thick and curly but sharp soap was formed, but as soon as the resin and the required quantity of the remaining lye were added, the soap boiled more easily. The finished soaps were pale yellow and possessed considerable detergent properties. The necessary corrections were only made when everything boiled uniformly in the kettle, and were generally limited to the addition of a few bucketfuls of weak salt lye to the thick soap.

Soaps like the above are still made at the present time, but since tallow and lardaceous fats, cotton-seed oil, etc., are cheaper than palm-kernel oil, other combinations are made which require a different process of boiling. With only 10 to 15 per cent. of resin one may even venture to take one-half combining fats and one-half other fats.

A formula of 100 to 150 pounds of resin, 300 pounds of tallow, 200 pounds of cotton-seed oil, 400 pounds of palm-kernel oil, and 100 pounds of cocoanut-oil gives a fair quality of soap. This combination can also be advantageously worked by direct boiling, since the resin effects the combination and easy boiling. The process is the same as for the preceding, except that lye about 1 degree weaker is used.

The formula can also be treated indirectly by first boiling the tallow to grain, and it will be found that when this grain is thoroughly boiled and finally brought into the kettle, the soap acquires a beautiful appearance, which is still improved by storing.



*Oranienburg curd-soap*.—The so-called Oranienburg curd-soap is a smooth resin curd-soap. It being generally demanded of a light, clear color, the palest kinds of French or American resin only can be used, and only fats which yield clear white soaps, such as white palm-kernel oil, tallow, cotton-seed oil, white oleic acid, lard or lardaceous fats, light bone-fat, and, if the state of the fat and oil market allows, some bleached linseed-oil. It is advisable to use at least two-thirds palm-kernel oil and one-third of the above fats. In winter it is especially necessary to work at least one-third tallow, bone-fat, or lardaceous fats, as soaps from pure palm-kernel oil or boiled in connection with a little cotton-seed oil are inclined to mould. The following are suitable formulæ :—

*For winter*: White palm-kernel oil, 600 pounds; tallow or pale bone-fat, 200; cotton-seed oil, linseed-oil, or pale oleic acid, 100; pale resin, 180 to 225 (or 20 to 25 per cent. of the fat used).

*For summer*: White palm-kernel oil, 700 pounds; tallow, lard, or bone-fat, 100; cotton-seed oil, linseed-oil, or oleic acid, 100; pale resin, 180 to 225.

Small deviations from the above prompted by the state of the market exert, of course, no great influence upon the result.

All waste, especially the same soap, resulting from salting out the precipitate of paste from a previous boiling, is brought into the kettle and boiled upon lye of at least  $29^{\circ}$  to  $30^{\circ}$  B. until all froth has disappeared, the quantity of lye required being about one-half of that of the fats to be used. The palm-kernel oil is then carefully introduced, the comminuted resin being held in readiness to promote combination, which generally takes place immediately after the addition of one-half of the resin. This also prevents the mass from rising too violently, lye being also held in readiness for the latter purpose. The greatest care is always required in combining fats with such strong lye.

Combination being induced and the mass boiling quietly and uniformly, the quantity of lye still wanting and the rest of the fats are gradually introduced until the soap boils thick and curly, but shows a perceptible touch. With a moderate fire, the rest of the resin is then allowed to dissolve and the mass again fitted to a good caustic taste. With an average lye of  $30^{\circ}$  B. the soap will now

scarcely boil too thick, because the more resin is worked in the thinner the soap becomes and the stronger must be the lye required towards the end of the operation. A slight excess of lye, perceptible upon the tongue or by dull, turbid streaks upon the spatula, suffices for the separation of a precipitate of paste. If, however, the soap should boil somewhat too thick and heavy, a few bucketfuls of water, weak lye, or salt water suffice to render it more loose and thinner. Light, loose boiling is an indication of the soap being finished. The fire is now removed, the kettle covered for a few hours, and the soap finally framed while hot. The frames remain covered for two days for the paste to settle.

The soap being ladled into the frames, but a small quantity of paste remains in the kettle, which, when boiled with oleic acid or bone-fat and salted out, yields very little sub-lye. The more viscid and fatter such precipitate of paste is—which is attained by avoiding salt as much as possible and not fitting too fine—the more can one count on a good settling in the frames. Such precipitate of paste, when cold, can be drawn like leather from the curd-soap adhering to it, which is pure and salable close to the precipitate.

Soap boiled according to the above-described process will never show froth, as its formation is prevented by the use of sufficiently strong lye. Hence it is far better to be obliged to add some water or weak lye towards the end of the boiling than to have to evaporate perhaps for hours, which, besides, does not always prevent the formation of froth. This method offers the further advantage of not having to drag large quantities of precipitate of paste from one boiling to the other, as is so frequently the case even in factories manufacturing large quantities of these soaps. And, besides, large boilings can be faultlessly finished in a comparatively short time over an open fire without the use of steam.

All soaps largely boiled from palm-kernel oil have a viscid but beautiful and lustrous formation of grain-fibre, and can only be regularly divided by a well-constructed cutting machine.

The yield, inclusive of the curd-soap recovered from the precipitate, and counting the resin as fat, is about 150 per cent., which could be increased by the use of larger proportions of tallow, palm-oil, or good lardaceous fats, and might turn out

somewhat less with the use of more liquid vegetable fats, such as linseed and cotton-seed oils.

*Yellow resin curd-soap.*—Yellow resin curd-soap, also called *palm-kernel oil soap* or *yellow curd-soap*, is prepared in the same manner as the preceding, with the exception that the soap is colored with 5 to 15 per cent. of crude palm-oil, according to the demand of customers. Where a dark color is not objectionable, larger quantities of dark fats, especially bone-fat, can be worked in; it being, however, advisable in this case first to boil the bone-fat to grain in order to purify it and modify its odor. A higher percentage of resin is also generally used. In fact, the soap is found in so many shades as to render it difficult to recommend any formula, since the soap-boiler has to be guided by the demands of his customers. Care must, however, be had not to use too much crude palm-oil, as the yellow lather caused by it would give rise to complaints. More than 12 to 15 per cent. of the fat used is decidedly objectionable, 5 to 10 per cent. being generally sufficient.



## CHAPTER XIII.

## HARD SOAPS (CONTINUED).

II. *Paste-Soaps.*

UNDER paste-soaps we understand products which, when finished and in a hot state, have the appearance of clear solutions of paste, but when cold form moderately firm soaps. It is always a good sign when the finished soap shows a slight froth, which in the covered kettle decreases to a thin film. In pouring out the soap more froth is generally formed, which cannot be prevented, and, besides, is not injurious, since the soap, if kept at a temperature of  $167^{\circ}$  F., purifies itself by expelling all foreign substances. The toilet-soaps known as glycerin-soaps, as well as the cheaper cocoanut-oil soaps, also belong to this class. They will, however, be described later on, since, being perfumed and colored, they are not intended for laundry use. Soaps intended for the latter purpose can only be colored without injury by such old-established coloring substances as Frankfort black, ultramarine, and bole. Yellow and brown, being also favorite colors for household soaps, are obtained by the fats and resin. For yellow, crude palm-oil and 5 to 10 per cent. of resin suffice, while for brown, the same materials intensified by some sugar color are used in case no fats giving the required coloration, such as wool fat, fuller's fat, or brown fats, are employed.

As regards their external appearance paste-soaps may be divided into two groups: smooth and marbled soaps. In respect to their other properties nothing definite can be said, since there are so many varieties that their number cannot even be approximately determined, the differences depending on the yield, which may vary from 250 to 1600 per cent. and on the fats used.

All these soaps can be prepared by the direct method by the use of weak lye. Their manufacture is, however, rendered en-

tirely safe by always treating the base-soaps as curd-soaps and augmenting them to the greatest yields by means of salt, etc. The soap-boiler who uses caustic lye at discretion can never foretell the yield he will obtain from the finished soap.

For the manufacture of paste-soap with a large yield, the use of double the quantity of caustic lye required for the complete saponification of the fats signifies nothing, because as soon as augmentation exceeds 260 per cent. caustic lye has to be added in order to obtain soap of sufficient hardness on the cut surface. It is different, however, with soaps of a low yield, which are to resemble curd-soaps or at least half-grain soaps. This can be effected by taking only as much caustic lye as is required for the saponification of the fats and using the materials for augmentation in as concentrated a form as possible.

Three kinds of augmenting materials are generally used: (1) Lyes containing carbonates and common salt, which is the easiest way of preparing paste-soap, (2) water-glass, and (3) flour, talc, clay, cryolite, each by itself, or in connection with solution of common salt, lye containing carbonate, or water-glass.

*Paste-soaps with a yield of 250 to 275 per cent.*—Paste-soap to be an imitation of curd-soap must resemble it in its principal properties, dry out but little, and be economical in use, the attainment of which depends partially on the fats used and partially on the filling material.

The best smooth white or colored soaps, which, if desired, can be artificially marbled, give a yield of about 275 per cent.

The following may serve as a formula for such soap:—

100	pounds of cocoanut-oil,
50	“ tallow,
75	“ caustic soda lye of 36° B.,
60	“ water-glass, mixed with
45	“ tank-lye of 12° B.,
5	“ common salt, dissolved in
20	“ water,
60	“ tank-lye of 5° B.

The process is as follows:—

The tallow and cocoanut-oil melted and heated to 189.5° F. are stirred together with the 60 pounds of tank-lye of 5° B.,

previously mixed with about 40 pounds of the caustic soda lye of  $36^{\circ}$  B. As soon as combination is effected the remaining 35 pounds of the caustic soda lye are added. When the combination forms a solid soap, and the latter, which is kept at a temperature of from  $189.5^{\circ}$  to  $201^{\circ}$  F., shows an inclination to thicken, the salt water is added. Even an actual thickening would signify but little, and would be a sure sign of the establishment of an actual combination between the fats and the lye. In order to remedy this defect the soap should be kept right hot and crutched until the pieces are dissolved. When this is the case the water-glass mixed with lye is added. After again crutching for some time the kettle is covered and samples are taken later on. If the samples prove not sufficiently solid, caustic lye has to be added in summer, and in winter a solution of about three pounds of common salt in water. A correct fit is readily effected and learned by a few experiments.

Soaps thus prepared keep very well if not hardened too much either by means of salt water or caustic lye, and they will never petrify in consequence of the water-glass they contain.

By substituting for the 60 pounds of water glass in the above formula 30 pounds of water-glass and 40 pounds of talc, the proportions of salt and lye remain the same, but the execution of the process varies somewhat. The 40 pounds of talc are crutched into the hot fats, and after effecting saponification in the same manner as above with 60 pounds of tank-lye of  $5^{\circ}$  B. and 75 pounds of caustic soda lye of  $36^{\circ}$  B. and combination is established, the 25 pounds of salt water are added, and finally the 30 pounds of water-glass mixed with 25 pounds of tank-lye of  $12^{\circ}$  B. After testing the finished soap is treated in the same manner as the preceding.

The formula may also be changed by substituting palm-kernel oil for a portion of the cocoanut-oil, or by using palm-kernel oil alone. As long as more cocoanut-oil is used than any other fat, for instance, 60 pounds cocoanut-oil, 40 pounds palm-kernel oil, and 50 pounds tallow, the proportions of lye and salt may remain the same, though great care must be had in fitting. If, however, the quantity of cocoanut-oil is less than half of that of palm-kernel oil, the strength of the caustic lyes should be less



by 1 to  $1\frac{1}{2}^{\circ}$  B. With the use of palm-kernel oil alone it is advisable to change the formula to 110 pounds of palm-kernel oil and 40 pounds of tallow and caustic soda lye of  $35^{\circ}$  B. instead of  $36^{\circ}$  B.

For tallow may also be advantageously substituted other fats containing stearin, especially crude or bleached palm-oil. With the use of sedimentary oils from oil-reservoirs, or cotton-seed oil or peanut-oil, the resulting soaps though serviceable are less solid.

Soaps of an equally good quality as the preceding are those brought into commerce under the name of yellow soap, or filled resin-soap, with a yield of 275 per cent.

The formula differs from the preceding one in so far that it consists of more or less cocoanut-oil, palm-kernel oil, crude palm-oil, and resin.

Such a formula, for instance, would consist of:—

90	pounds of cocoanut-oil,
10	“ crude palm-oil,
20	“ dark resin,
60	“ caustic soda lye of $36^{\circ}$ B., mixed with
40	“ tank-lye of $3^{\circ}$ B.,
40	“ water-glass, mixed with
10	“ caustic lye of $30^{\circ}$ B.,
2	“ common salt, dissolved in
10	“ water.

The process is apparent from the formula itself. After melting the fats and the resin, the 60 pounds of caustic lye of  $36^{\circ}$  B. and the 40 pounds of tank-lye of  $3^{\circ}$  B. are crutched in, and a good combination being established the salt water is added and finally the water-glass. The soap does not require boiling, but must be kept at a temperature of above  $189.5^{\circ}$  F. Small samples of the finished soap must be firm and hard; if otherwise, fitting is wanting, and the defect is remedied by adding a few pounds of caustic lye of  $36^{\circ}$  B. and again heating until all is thoroughly combined. Too much sharpness must, however, be avoided, since this would frequently result in stained soaps.

This variety of soaps also embraces all other soaps with a yield of about 250 per cent. and which, though the finished product shows all the characteristics of a grain or half-grain

soap, are formed into smooth soaps partially by the method of their augmentation and partially by the manner of their framing. The base-soap for these soaps is also either a grain or half-grain soap to which are added as condensed solutions as possible.

The principal condition for such soaps is that with a correct and moderate fit the fats used should yield a product dry and solid on a cut surface. A very solid grain-soap can, for instance, be boiled from bone-fat by using much salt and thoroughly evaporating, but by augmenting such soap the yield would not exceed 170 to 180 per cent., as the soap would either break up or become too soft. Such fats used by themselves are not suitable for filled soaps. Tallow or bleached palm-oil are better adapted for the purpose, and 10 to 20 per cent. of resin can without injury be added to such soaps. Green sulphur-oil (olive-oil extracted from the press-residue) gives also a firm grain-soap which can be subsequently augmented.

The above-mentioned three varieties of fats are the only ones, which boiled, either by themselves or in connection with each other, yield grain-soaps allowing of a regular and paying augmentation. The preparatory boiling to grain may be executed in the customary manner, provided the final result be a solid, lean soap, the other properties of the grain, whether it be dull or sharp, contributing nothing to the successful filling of such soaps with lyes containing carbonate and common salt. Solution of carbonate of  $10^{\circ}$  to  $12^{\circ}$  B. heated to from  $167^{\circ}$  to  $189.5^{\circ}$  F. can be either crutched into such grain-soaps or the whole may be allowed to boil up and the paste formed hardened with salt water. The result is a genuine filled soap and the yield can be considerably increased.

It is, however, different if filling agents such as water-glass and talc are to be used. The base-soap must then be as neutral as possible, which can only be attained by the grain showing good saponification and being subsequently ground until any excess of sharpness has subsided.

Water-glass of  $50^{\circ}$  B. can be simply crutched into correctly prepared base-soaps of this kind, and with a yield of 250 per cent. they will remain thick-ribbed similar to grain-soap. There being a difference in the various kinds of water-glass, it may be

sometimes necessary to assist with filling material containing more water, talc mixed with water or simply hot water being frequently added if the soap with a yield of 250 per cent. is very thick.

Should the base-soap not take up the water-glass, *i. e.*, tear so as to appear as if salted out, the cause of this defect may be due to the base-soap containing too much water, salt, and lye, or to the water-glass itself, it being extremely difficult even for the most expert soap-boiler to form a correct judgment. It is well known that water-glass rich in silicic acid contracts every kind of grain-soap, and only forms paste-soap which will draw threads when the yield reaches 290 per cent., and then augmentation can be extended to about 340 per cent. On the other hand, with weak water-glass containing little silicic acid, for instance, water-glass diluted to 20° B., the same good grain-soap would in many cases completely salt out with a yield of 200 per cent. Hence, if a grain-soap contains from the start much fixed water, which in this case even holds salt and soda in solution, the base-soap will not fill but separate.

What is a defect in good grain-soaps becomes, however, a necessity in other grain-soaps boiled from tallow in connection with cocoanut-oil or palm-kernel oil. Grain-soap made with 50 per cent. of cocoanut-oil can seldom be filled with strong water-glass alone, an addition of salt water, soda solution, or even caustic lye being generally necessary. Moreover, every soap requiring salt or lye in filling with water-glass in order to preserve the latter intact, becomes rough and hard in drying. To avoid such additions it is only necessary to add pure water or dilute solutions of water-glass as may be necessary, the required salts being then spontaneously formed.

*Tallow soda-soap.*—The soap known under this name is generally of a brilliant white color. It is poured into small low frames, curly flowers being drawn into the surface with a rod. It is generally prepared from two-thirds palm-kernel or cocoanut-oil and one-third tallow, and filled with solution of carbonate of soda or with water-glass. The best qualities are prepared from two-thirds tallow and one-third cocoanut-oil and a yield of 200



to 220 per cent., while that of the poorer varieties is 300 per cent.

The soaps can be boiled by the direct method, as is frequently done for convenience sake. To obtain the best quality possible, it is, however, advisable first to boil the tallow and also a portion of the other fats to grain so that the finished soap will resemble a filled grain-soap rather than a paste-soap.

The following may serve as a formula for tallow soda-soap by the direct method: Palm-kernel oil 100 pounds, tallow 50 pounds, caustic soda lye of 35° B. 80 pounds, potash solution of 25° B. 100 pounds, salt water of 20° B. 50 pounds.

The fats are melted and passed through a fine sieve, it being also necessary to purify the lye and salt water in the same manner and to scour the kettle bright, as the soaps with a yield of 250 per cent. are, as a rule, not sufficiently thinly fluid to expel fine particles of dirt.

The 80 pounds of caustic soda lye of 35° B. are mixed with 10 pounds of water and 30 pounds of the potash solution of 25° B. With the resulting lye the fats are saponified and the lye is kept at a temperature of 201° F. As soon as combination is established, the salt water is gradually added and later on the remaining 70 pounds of potash solution of 25° B. The kettle is then well covered for about one hour, after which the soap is thoroughly crutched and tested. A sample should be hard, not lardaceous to the touch and pliant; if otherwise, fitting is wanting, which is effected in summer by adding a few pounds more of caustic soda lye and perhaps a few pounds of salt water, but in winter by salt water alone so as to avoid moulding of the soap. After the addition of lye or salt water the soap is again heated.

When a sample shows the soap to be sufficiently solid, it is again covered and a moderate coal fire kept up under the kettle to disperse the froth. The slight coat of froth which finally remains is lifted off, and after the clear soap is brought into low sheet-iron frames, the surface is drawn curly with a rod, and the soap left standing uncovered. In about eight hours it is ready for cutting. The brilliantly white bars are dried in the air and provided with the factory stamp.

The same combination of fats can also be converted into soap by

indirect boiling and filled with water-glass or tale. Such a formula would be about as follows: 100 pounds of tallow are boiled to grain with 240 pounds of caustic soda lye of  $13^{\circ}$  B., while 200 pounds of palm-kernel or cocoanut-oil are boiled to a paste-soap with 135 pounds of caustic soda lye of  $35^{\circ}$  B., 60 pounds of water-glass, 60 pounds of water, and 5 pounds of common salt.

The process is carried out by first boiling the grain, and after emptying the kettle introducing at once the 135 pounds of caustic soda lye of  $35^{\circ}$  B., the water, and the salt. After heating this mixture to  $189.5^{\circ}$  F., the water-glass is added, and the whole thoroughly crutched. The warm grain is then added, and the previously melted palm-kernel or cocoanut-oil crutched in. Combination takes place by keeping the soap at from  $189^{\circ}$  to  $200^{\circ}$  F., a quite thinly fluid soap free from froth being formed. The soap is not covered, but slowly crutched until a sample shows it to be sufficiently solid. If too soft, it is hardened by adding a few pounds of caustic soda lye. It might, however, be possible that evaporation has been carried too far and the resulting soap be brittle and rough. In such a case add hot water holding a little salt in solution.

→ *Paste-soaps with a yield of 300 to 350 per cent.*—The manufacture of these soaps offers less difficulty than that of the preceding as long as only smooth or simply colored soap is to be prepared. However, the marbled variety known as English mottled soap or *Eschweg III.* also belongs to this class.

The simplest way of preparing these soaps is by using a formula consisting of palm-kernel or cocoanut-oil 100 pounds, caustic soda lye of  $36^{\circ}$  B. 60 pounds, potash solution of  $25^{\circ}$  B. 100 pounds, salt water 60 pounds.

The process is easy and sure, though it is necessary, as is the case with every other kind of soap, to adhere to the principle of not adding the salt solutions until the fats and lye are thoroughly combined. The formula may also be so changed as to use tallow or fats in connection with palm-kernel or cocoanut-oil. As much as one-half the quantity of tallow or palm-oil may be used; of soft fats it is, however, advisable not to exceed 30 parts to 70 parts of cocoanut-oil, and of still softer or liquid fats 20 parts to 80 parts of cocoanut-oil. The other proportions remain the

same, though the strength of the caustic lyes is best reduced  $1^{\circ}$  to  $1\frac{1}{2}^{\circ}$ .

With these soaps much talc is used as a filling material. The fats are heated to  $189.5^{\circ}$  F., the talc crutched in, and the lye added. The following is a suitable formula: Cocoanut-oil 100 pounds, talc 50 pounds, caustic lye of  $36^{\circ}$  B. 60 pounds, potash solution of  $25^{\circ}$  B. 80 pounds, salt water of  $20^{\circ}$  B. 50 pounds.

The following formula, which is almost the same as the above only that water-glass is added, might be preferable as the soap keeps better:—

100	pounds of cocoanut-oil,
50	“ talc,
60	“ caustic soda lye of $36^{\circ}$ B.,
40	“ water-glass, mixed with
10	“ caustic soda lye of $36^{\circ}$ B.,
3	“ common salt, dissolved in
10	“ water,
50	pounds of tank-lye of $3^{\circ}$ B.

The operation is carried on as follows: The talc is stirred into the fats heated to  $189.5^{\circ}$  F. While keeping the mass at this temperature, the 60 pounds of caustic soda lye of  $36^{\circ}$  B., previously mixed with the 50 pounds of tank-lye of  $3^{\circ}$  B., are crutched in. When a thorough combination is formed, the salt water is added and then the water-glass solution. Samples are now taken, and if they are hard the kettle is covered to allow the froth partially to disperse and partially collect on the top. The portion of the froth collecting on the top is then lifted off and the whole crutched for some time to prevent the talc from settling. The soap is finally brought, at a temperature of about  $156^{\circ}$  F., into low frames. To promote cooling any cuttings of the same soap which may be on hand are added to the hot soap in the frames.

By substituting in the above formula palm-kernel oil for cocoanut-oil, the soap may be prepared by first boiling the fat to grain. The filling, which consists also of talc and water-glass, is finally crutched into the grain-soap. This gives a milder product, no lye being added to the water-glass. Such a formula consists of:—



100 pounds of	palm-kernel oil, boiled with
100	“ caustic soda lye of 25° B.,
50	“ talc,
30	“ water-glass, mixed with
20	“ water,
30	“ salt water of 5° B.

Ninety to 95 pounds of the caustic soda lye of 25° B. are gradually crutched into the palm-kernel oil heated to 189.5° F. The reason for reserving 5 to 10 pounds of the lye is because as the action of the lye varies very much, the quantity given in the formula may be too large. When a thorough combination is established, 4 pounds of common salt dissolved in 8 pounds of water are added and the mass is kept at a boiling heat, though actual boiling is not necessary. The object is attained if the soap separates in the manner of grain-soap upon a precipitate of paste; otherwise caustic lye of 25° B. is crutched in until separation takes place. The settled grain-soap is ladled into a barrel, and after removing the sub-lye returned to the kettle. The liquor resulting from stirring the 50 pounds of talc into the 30 pounds of hot salt water of 5° B. is then heated to from 144.5° to 167° F. and crutched into the grain. When the whole forms a thoroughly combined soap, the 30 pounds of water-glass mixed with the 20 pounds of water and heated to 144.5° F. are gradually added.

*English mottled soap.*—This soap, also called *Eschweg soap III.*, is also a filled soap, which is, however, marbled by the formation of flux. This variety of soap is best formed in frames holding 1200 to 1600 pounds. The following is a suitable formula :—

400 pounds of	palm-kernel oil,
200	“ cocoanut-oil,
300	“ caustic soda lye of 36° B., diluted with
200	“ water,
72	“ common salt, dissolved in
144	“ water,
120	“ potash and
30	“ calcined soda, dissolved in
600	“ water,
2	“ ultramarine, stirred into
16	“ water, and
30	“ water-glass added thereto.

The 400 pounds of palm-kernel oil and 200 pounds of coconut-oil are melted and saponified with the 300 pounds of caustic soda lye of  $36^{\circ}$  B., previously diluted with the 200 pounds of water, the mass being kept at a temperature of  $189.5^{\circ}$  F. As soon as combination is effected, some of the salt water is added and later on the solutions of soda and potash. The kettle is then covered and the soap, which should show a temperature of about  $189.5^{\circ}$  F., is allowed to stand for one hour, when the base-soap will be quite free from froth. It is then thoroughly crutched and tested as to whether it will draw threads. If it drops from the spatula it is too weak, which is remedied by adding a few pounds of caustic lye of  $30^{\circ}$  B., again heating and crutching.

When the base-soap is uniformly warm and bright, about 20 to 30 pounds of it are taken from the kettle.

The ultramarine and water-glass are mixed together cold by first dissolving the color in the water and then adding the water-glass.

In order to test whether the base-soap is adapted for coloring and marbling, some of the coloring mixture is mixed with the 20 to 30 pounds of soap taken from the kettle. If the sample acquires a uniform blue color the base-soap is so alkaline as to render it impossible for the water-glass to crystallize out. By now adding to this colored sample 200 to 400 pounds of melted palm-kernel oil and stirring it uniformly through the soap, the oil withdraws from the latter so much sharpness that the coloring substance separates in large flakes, which form the marbling. If the soap has a suitable temperature, about  $158^{\circ}$  F., it is so thickly fluid that the marbling formed cannot subside, but remains uniformly distributed throughout the soap. As soon as this is established with the sample of 20 to 30 pounds, the soap in the kettle is allowed to stand covered for some time. If after-heating takes place the flux, as a rule, subsides and has to be again crutched up, it being reformed as long as the soap is hot enough. For soaps inclined to after-heating it is best to let them stand in the open frames, which should not be made too full. If the coloring substance added to the sample of soap does not distribute itself in it, but crystallizes out, the base-soap is not sufficiently alkaline. To remedy this, either lye is added to the soap or, if but little is

wanting, a few pounds of caustic lye are added to the coloring substance and a fresh test is made. All samples no longer suitable for testing are returned to the kettle and fresh ones taken.

In mixing the coloring substance with the sample it may happen that broad streaks are formed, which, after a short rest, contract to a large, broad marbling. This, as a rule, is the most successful soap and need not be covered.

This test with a small sample is absolutely necessary before bringing the soap into the frames. When it is found that the soap is suitable for coloring, it is brought into the frames at a temperature of from  $167^{\circ}$  to  $174^{\circ}$  F., and, after covering the frame for a short time to allow the froth to disperse, the coloring substance is crutched into the frame, and, if necessary, the melted palm-kernel oil. All observations and changes made in manipulating the small samples are followed up on the soap in the frame. Any spoiled soap is re-melted together with any cuttings on hand, and again brought in the frames. Much patience and considerable experience are required for the successful manipulation of this kind of soap.

*Resin-paste soaps.*—The favorite of these soaps is the pale-yellow variety with a yield of 330 to 350 per cent. A suitable formula consists of:—

180	pounds of	cocoanut-oil,
20	“	crude palm-oil,
40	“	resin,
140	“	caustic soda lye of $36^{\circ}$ B.,
180	“	water,
80	“	water-glass, mixed with
20	“	caustic soda lye of $36^{\circ}$ B., diluted with
4	“	common salt, dissolved in
16	“	water.

The resin is dissolved in the melted cocoanut and palm-oil. The caustic lye diluted with the water is then added, and the whole kept at a boiling heat, even actual boiling doing no harm. The salt water is then added, and the mass kept at the same temperature for one to two hours. The water-glass mixed with the caustic lye is finally added, and the kettle allowed to stand covered for at least one hour. The mass is then thoroughly crutched and a sample taken. Should the soap prove too soft, it is hardened with caustic soda-lye.



These soaps must, however, contain neither too much lye nor too much salt, as otherwise they turn out moist and mottled. If, therefore, the sample is any way solid, it is best not to add any more caustic lye, but to dry the cut soap. If a higher color is desired, 30 to 40 pounds of crude palm-oil may, without injury, be used instead of 10 pounds.

The following formula is nearly the same, talc being substituted for water-glass as filling material :—

160	pounds of	cocoanut-oil,
40	“	crude palm-oil,
40	“	dark resin,
140	“	caustic soda lye of 36° B., diluted with
160	“	water,
100	“	talc,
6	“	common salt, dissolved in
34	“	water.

The 100 pounds of talc are stirred into the melted fat and resin, and the whole saponified with the caustic lye, after which the salt water is added. The only difference in the manipulation of this soap from the preceding one is, that it should not be allowed to boil, but kept at a temperature of about 189.5° F. When a sample shows the soap to be sufficiently solid, it is ladled into the frames and crutched to prevent the talc from settling. For a finer quality of soap filled only with solutions of potash and common salt, the following formula may be used :—

140	pounds of	cocoanut-oil,
40	“	tallow,
20	“	crude palm-oil,
40	“	pale resin,
140	“	caustic soda lye of 36° B., diluted with
60	“	water,
160	“	potash solution of 30° B.,
80	“	common salt solution of 22° B.

This soap does not require boiling, being only kept at a temperature of 189.5° F. After dissolving the resin in the melted fat, the liquid mass is passed through a hair-sieve, returned to the kettle, and saponified with the caustic lye. When combination is established, the solutions of common salt and potash are gradually added. A sample taken from the kettle should show a consider-

able degree of solidity, otherwise the resulting soap will be too soft. If necessary, a few pounds of crystallized soda, and, perhaps, a little caustic lye, are added, the soap being again heated after each addition. After frequent crutching, the soap, at a temperature of from  $144.5^{\circ}$  to  $156^{\circ}$  F., is brought into low frames.

The color of brown resin-paste soaps is due to the dark resin and the fats used. The following may serve as a formula :—

150	pounds of	cocoanut-oil,
50	“	wool fat,
40	“	dark resin,
140	“	caustic soda lye of $36^{\circ}$ B., diluted with
140	“	water,
80	“	water-glass, mixed with
20	“	caustic soda lye of $36^{\circ}$ B.,
6	“	common salt, dissolved in
14	“	water.

The process of manipulation is the same as for the preceding soap. In case the soap should not turn out dark enough, it is colored with 4 pounds of sugar color dissolved in potash solution.

The yield of these soaps may be increased to 400 per cent. and more by the addition of carbonated lye, crystallized soda, or salt solution, 4 pounds more of caustic lye of  $36^{\circ}$  B. being calculated for every 20 pounds. Such soaps are, however, inclined to efflorescence, and are rendered hard and rough by the addition of a larger percentage of water-glass.

*Paste-soaps with a yield of 500 to 600 per cent.*—The following soaps with a yield of 500 to 600 per cent. are prepared either smooth or marbled. The smooth soaps are allowed to retain their natural color as derived from the fats used in their preparation, or are artificially colored, while the marbled soaps are partially artificially mottled and partially so boiled as to become mottled by the formation of flux.

The following is a formula for smooth soaps :—

160	pounds of	palm-kernel oil,
40	“	cocoanut-oil,
120	“	caustic soda lye of $36^{\circ}$ B., diluted with
120	“	water,
50	“	potash and
50	“	calcined soda, dissolved in

200	pounds of water,
120	“ salt, dissolved in
200	“ water,
40	“ caustic soda lye of 36° B. for fitting.

Combine the 200 pounds of oil with the 120 pounds of caustic lye of 36° B., previously diluted with the 120 pounds of water at 189.5° F. By adding the solution of common salt too soon, combination is rendered difficult and sometimes almost impossible, and all subsequent defects of the soap can be traced to faulty saponification. After adding the solutions of common salt and of potash and soda, keep the soap at a boiling heat, and only if a sample of it should not prove solid enough add gradually a sufficient quantity of the 40 pounds of caustic lye of 36° B. prescribed for fitting.

It may be necessary, in order to obtain the soap of sufficient hardness, to use even more than the 40 pounds of caustic lye of 36° B. prescribed, which may be due to the lye not being caustic enough, as the caustic soda frequently contains much carbonate. In this case note how much lye is used for saponification and subsequent hardening and take 10 to 12 pounds less of calcined soda for a subsequent boiling. The soap is brought into the frames at a temperature of 167° F., covered for one hour, and then let stand uncovered.

These soaps may be colored red with bole, colcothar, or cinabar. To color them yellow, substitute about 20 pounds of crude palm-oil for 20 pounds of palm-kernel oil and add about 10½ ounces of potassium bichromate, dissolved in hot water, to the soap. A brown color is obtained with sugar color.

A similar formula with talc filling is as follows:—

40	pounds of cocoanut-oil,
160	“ palm-kernel oil,
100	“ talc,
120	“ caustic soda lye of 36° B., diluted with
120	“ water,
80	“ potash and
40	“ crystallized soda, dissolved in
200	“ water,
100	“ common salt, dissolved in
160	“ water,
40	“ caustic soda lye for fitting.



The soaps filled with talc are prepared in the same manner as the preceding; they are, however, brought into the frame at a temperature of  $167^{\circ}$  F. and crutched until they are cold and thick to prevent the talc from settling. It is advisable to remove any froth formed in the frame, as it is apt to form stains in the clear soap. These soaps are also colored.

A third formula with water-glass is as follows :—

160	pounds of	palm-kernel oil,
40	“	cocoanut-oil,
120	“	caustic soda lye of $36^{\circ}$ B., diluted with
120	“	water,
100	“	water-glass, mixed with
20	“	caustic soda lye of $36^{\circ}$ B.,
40	“	crystallized soda, and
20	“	potash, dissolved in
200	“	water,
20	“	common salt, dissolved in
60	“	water,
60	“	caustic soda lye of $36^{\circ}$ B. for fitting.

The process is the same as for the preceding soaps. Combine the fats with the caustic soda lye, add the solutions of common salt and of potash and crystallized soda, and after boiling the whole thoroughly, add the water-glass and take samples. If the soap is not solid enough, add as much caustic soda lye as is required.

The last variety of these soaps is that filled with talc and water-glass, which, on account of the filling material employed, dries out least of all. The following formula may be used :—

160	pounds of	palm-kernel oil,
40	“	cocoanut-oil,
120	“	talc,
140	“	caustic soda lye of $36^{\circ}$ B., diluted with
140	“	water,
120	“	water-glass, mixed with
40	“	caustic soda lye of $36^{\circ}$ B.,
40	“	crystallized soda and
20	“	potash, dissolved in
160	“	water,
20	“	common salt, dissolved in
100	“	water,
40	“	caustic lye of $36^{\circ}$ B. for fitting.

The talc is stirred into the melted fats and the latter are saponified with the caustic lye at  $189.5^{\circ}$  F. When a thorough combination is established the salt water and solutions of soda and potash are alternately added and finally the solution of water-glass. The soap is kept at  $189.5^{\circ}$  F. and fitted with caustic soda lye. If correctly fitted, this soap is more thickly fluid than the preceding ones and hence can be brought hotter into the frames, where it is, however, somewhat crutched.

This variety of soap is also adapted for artificial marbling, which is best effected by taking a portion of the soap from the kettle, heating it right hot, and coloring it with coloring matter stirred in water or oil. The soap remaining in the kettle is then cooled to about  $133^{\circ}$  F. by crutching. A layer about  $1\frac{3}{4}$  to  $2\frac{1}{4}$  inches thick is then brought into a low frame; upon this are poured thin rows of the colored soap, which are drawn and crutched in so as to form colored streaks. This is alternately continued until the frame is filled, when the soap is allowed to stand uncovered. The more skilfully this manipulation is executed the better the appearance of the soap.

Soaps with a yield of 500 to 600 per cent. can also be marbled by the formation of flux. The following may serve as a formula:—

200	pounds of	palm-kernel oil,
140	“	caustic soda lye of $36^{\circ}$ B., diluted with
200	“	water,
60	“	potash and
60	“	calcined soda, dissolved in
200	“	water,
140	“	common salt, dissolved in
140	“	water.

The base-soap is prepared like the preceding, and, as a rule, requires no lye for fitting; but if the soap is too soft, caustic soda lye of  $36^{\circ}$  B. is added.

The soap is brought into the frame at a temperature of  $167^{\circ}$  F. and the coloring substance, stirred in water, crutched in. For the above formula about 10 ounces of ultramarine and  $3\frac{1}{2}$  ounces of Frankfort black suffice for a blue-gray color. The coloring material is mixed with about 10 pounds of hot water and 10 pounds of water-glass, about 10 pounds of tank-lye of  $20^{\circ}$  B.

being finally added to the mixture. After the coloring substance is uniformly distributed throughout the soap and the latter cooled off to about  $149^{\circ}$  F., about 10 pounds of melted palm-kernel oil are added and the whole thoroughly crutched. In about half an hour, during which time the frame remains covered, a large, broad flux will have formed. Should the soap have again become heated, which is determined with the thermometer, the flux, as a rule, subsides. The whole must then again be crutched up, and this operation, if necessary, repeated.

*Liverpool-soap.*—Under this name a yellow soap is manufactured with a yield varying between 300 and 800 per cent. This soap was originally prepared from crude palm-oil, the dirty sedimentary oils being used. Paste-soaps with a yield of about 360 per cent. possess the peculiarity of purifying themselves by expelling all particles of dirt, the heavier and coloring substances falling to the bottom, and the lighter ones forming a coat of froth on the top.

Crude palm-oil was formerly brought into commerce in an entirely impure state and had to be melted and clarified, but at the present time almost all oil is purified before it reaches the market. The sedimentary oils form, however, still an article of commerce, especially in England, and generally serve for the fabrication of the yellow Liverpool-soap.

Crude palm-oil enters into a thorough combination with caustic lye, and the resulting soap is quite solid with a yield of 400 per cent.

The manufacture is effected by two different methods. The sedimentary oil is boiled either upon strong salt-lye, so that the grosser dirt subsides, or it is directly boiled to a soap which is made to yield 400 per cent. by the addition of carbonates. As grain-soap boiled from the oil contains considerable dirt, it is dissolved in carbonated lye of about  $10^{\circ}$  B. and hardened with salt. About ten per cent. of resin is also frequently used in connection with the oil, the resulting soaps yielding a more abundant lather.

*Transparent resin paste-soap.*—This name is given to soaps which appear transparent on the edges of the bars. This is effected by the fats used and an addition of alkalies. A favorite



formula is as follows: Ceylon cocoanut-oil 180 pounds, crude palm-oil (Lagos) 20 pounds, pale resin 40 pounds.

The fats are saponified with caustic soda lye of  $22^{\circ}$  B., to which about 25 per cent. of potash solution of  $28^{\circ}$  B. is added. The transparency depending on the addition of potash lye, only lyes and fats as pure as possible should be used, and the soap hardened only when it boils perfectly clear. For hardening salt water of  $24^{\circ}$  B. is used, though potash water-glass can be substituted for a portion of the salt. The generally very thick water-glass is diluted with potash lye of  $2^{\circ}$  B. until the solution shows  $20^{\circ}$  B., and is then alternately added with the salt water. A sufficient degree of hardness and permanent transparency is ascertained by repeated examination of samples. The soap when correctly prepared retains its transparency and is elastic on the cut surface, but later on becomes harder. By the use of crystallized soda for hardening the transparency suffers somewhat, though it may suffice in some cases. It is recommended to guard against the use of too much common salt.

*Transparent resin paste-soap (another method).*—First prepare a lye, not too caustic, of  $30^{\circ}$  B. Then combine in the kettle by slow boiling 70 pounds of cocoanut-oil, 30 pounds of crude palm-oil, 20 to 25 pounds of resin with about 30 pounds of the lye of  $30^{\circ}$  B., and then stir in with a more moderate fire, so that the mass does not boil, 50 pounds of the lye of  $30^{\circ}$  B. The soap should show a strong touch. Heating is then discontinued, and 20 pounds of carbonate of potash solution are sprinkled over the soap and crutched in, the result being a strong paste. Now add with constant stirring 70 pounds of water-glass, previously diluted with 3 pounds of alcohol and 3 pounds of lye of moderate strength, and cover the kettle for half an hour. Then ladle the soap into the frame, covering it well so that it will thoroughly crystallize.

## CHAPTER XIV.

## HARD SOAPS (CONTINUED).

III. *Half-Grain Soaps.*

THE fact that the manufacture of these soaps (called in Germany Eschweg soap) has been introduced wherever soap-factories exist, is a proof that the product is better than its reputation. The cheap price at which the soaps can be sold is not so much due to the product itself as to the easy manner of boiling and the consequent fabrication of enormous quantities.

Half-grain soaps can only be prepared with the assistance of cocoanut-oil or palm-kernel oil. They are boiled by either one of three methods: (1) *indirectly*, by a preparatory boiling to grain of a portion of the fats, (2) *directly*, by boiling all the fats together, and (3) from base-soaps, by first boiling the fats to a grain-soap, which is then augmented, as far as its consistency will permit, with carbonated lye, common salt, and water-glass.

*Half-grain soaps by the indirect method.*—The manufacture of these soaps consists chiefly in a preparatory boiling to grain of a portion of the fats, the object being the removal of coloring matter frequently adhering to poorer qualities of fat. Boiling by the indirect way being the easiest method of obtaining a beautiful product it is advisable for soap-boilers wishing to introduce these soaps to become proficient in it before attempting the manufacture by direct boiling from palm-kernel oil in connection with lighter fats, such as linseed-oil and cotton-seed oil, and augmenting the soap with water-glass and talc.

The quality of half-grain soap is intimately connected with its appearance, it being by no means insured by the use of the best fats, such as tallow, palm-oil, and cocoanut-oil. Such soap may show defective formation of flux and faulty consistency, become

tray-shaped in drying, lose much in weight, and wash away rapidly. It may be wanting in firm combination, a defect frequently due to over-evaporation. Strongly evaporated half-grain soap must not be fitted too sharp nor forced too far with salts. The appearance of the soap will be best when one is enabled by a suitable combination of fats to prepare a firm lardaceous product which is frequently not attained even by the use of solid fats alone. The best formula for the purpose is to use for 100 pounds of lardaceous fat, by which is understood a composition of half tallow or palm-kernel oil and half bone-fat, lard, etc., 50 pounds of cocoanut-oil as a combining fat. If softer fats, such as horse-fat, cotton-seed oil, sesame-oil, peanut oil, etc., are to be worked, it is advisable to take only one-third of these fats to two-thirds of solid fats (tallow or palm-oil). Half-grain soaps are never boiled without the addition of a fat securing a solid combination, cocoanut-oil or palm-kernel oil serving for the purpose.

The following formula may serve to illustrate the execution of the operation: Bone-fat or lard 1000 pounds, tallow or palm-oil 1000 pounds, cocoanut-oil 1000 pounds.

The 1000 pounds of bone-fat or lard and the 1000 pounds of tallow or palm-oil are slowly boiled with about 1000 pounds of caustic lye of  $12^{\circ}$  B. until a thorough union is established. In case this requires a long time, so that much water evaporates, which would make the lye stronger and the process of combination difficult, 20 to 40 pounds of water are added. However, if a union is not effected by these means, the furnace door is opened or the steam shut off and the mixture allowed to stand until the fat and lye are observed to combine. Firing is then slowly continued and 50 pounds more of lye are added, and when these have thoroughly combined the addition of lye is continued until the paste becomes bright and thick and the soap, after a further addition of lye, commences to "wet." For the saponification of 200 pounds of fat about 400 pounds of caustic lye of  $12^{\circ}$  to  $13^{\circ}$  B. are, as a rule, required. The paste generally "wets" before all the lye has been added.

To separate the paste about 8 per cent. of common salt of the fat used is brought into the kettle simultaneously with the rest



of the lye. By assisting with the crutch the soap tears and rises up broken. After boiling for some time the soap is tested as to sharpness, and in case the sub-lye indicates such, it will also be always perceived in the soap, which boils thick, forces up the clear, round grain, and is of hard consistency. Such clear grain, which is generally formed by the evaporation of too much water, is not required for half-grain soap. If, now, water be poured over the soap, the grain is dissolved, the entire mass boils up high and with greater ease, and an augmentation of the grain takes place by the mass absorbing the greater portion of the sharpness previously observed. If the formation of such a grain is at once attained by direct boiling, the after-boiling and addition of water are omitted. The grain is now allowed to separate from the sub-lye, and, if there is but one kettle, it is ladled into barrels.

For the second operation—boiling to combination—about 100 pounds of tank-lye of  $24^{\circ}$  B. are brought into the kettle for every 100 pounds of cocoanut-oil. The lye used for the purpose is the first runnings obtained by treating calcined soda with lime. Any soap-cuttings on hand are dissolved upon the lye; otherwise it is allowed to boil up by itself. The grain-soap in the barrels is then added and the whole boiled thoroughly through upon the combining lye until a thick, clear grain is formed. The cocoanut-oil, with the exception of 100 pounds, is then introduced, the mass being kept at a gentle ebullition and moderately crutched to promote the uniform distribution of the cocoanut-oil and rapid combination. After boiling for some time longer the soap is tested. If it shows any sharpness the retained 100 pounds of cocoanut-oil are added; but if sharpness is wanting, some caustic lye of  $24^{\circ}$  B. is first introduced and boiled through with the mass, and then the cocoanut-oil. A low percentage of combining lye having been intentionally given, it may happen that too little sharpness is present if the grain is so far ground out as not to be able to retain an excess of sharpness. If, however, the above directions for boiling have been approximately followed, there is but one thing which may apparently be wanting in a regular half-grain soap, and that is water. To be sure, no direct, valid

information can be given in this respect, but the soap-boiler can confidently proceed with fitting, which is effected with salt water.

The cuttings and the grain having been boiled clear upon the combining lye and the cocoanut-oil crutched in with moderate boiling, it is not likely that a frothy soap will be formed at this stage. There may, however, be a lack of water due to evaporation; the soap may be thin, which indicates that water is wanting. To such soap add carefully salt water of  $5^{\circ}$  B., half cover the kettle and boil until the soap is bright; and the thicker it boils afterwards the more salt water (of  $10^{\circ}$  to  $15^{\circ}$  B.) must be used and the boiling continued with the addition of salt water until the soap breaks into beautiful light roses. A sample when placed upon the warm board covering the kettle should remain warm for some time, and after cooling have a uniform white appearance and break, not viscid, but like curd-soap. It may happen that after adding 5 to 6 per cent. of salt (of the quantity of cocoanut-oil used) there may show itself some sharpness and consequently a slight froth, which is removed by the addition of a few pounds of cocoanut-oil mixed with water.

It will frequently occur that these soaps lie thick and compact in the kettle. To such soap it is best to add salt water of  $5^{\circ}$  B. from the start and then proceed in the same manner as before. Further, the soap formed may be viscid and have a glassy appearance, though be thick and free from froth. This indicates lack of sharpness and that the soap needs caustic lye. Add first strong salt water of  $20^{\circ}$  B., and if the soap does not change after the addition of about four per cent. of salt, add caustic lye of  $20^{\circ}$  B., but not too much, so that salt water may be subsequently used.

Half-grain soaps prepared with the use of cocoanut-oil can bear much common salt, but the exact quantity cannot be given, as it depends on the content of alkalies in the soap. They also demand alkaline carbonates for augmentation in order to keep them free from froth. The addition of salt water serves for hardening, which otherwise would have to be effected by means of caustic lye. In summer even a large quantity of carbonates in the soap does not matter, but in winter it is advisable to decrease the quantity of carbonates as much as possible and add

salt instead in order to prevent efflorescence. The yield obtained is satisfactory and could not be increased by another method of boiling nor by such filling agents as talc and water-glass. The soap should not be boiled down too much and too firm, but when boiling easily and breaking into roses over the entire surface is brought into the frames and crutched until cooled to  $167^{\circ}$  F. The frames are then lightly covered. If in the course of three hours layers of flux lying closely alongside each other are observed to form, the frame is uncovered and the soap allowed to cool completely.

By omitting the cocoanut-oil from the above formula and using palm-kernel oil alone, the behavior of the soap during boiling differs somewhat from the preceding, as palm-kernel oil does not form as strong a combination as cocoanut-oil and cannot stand as much alkali and salt for augmentation. The process of boiling is the same as the preceding up to the point of fitting and shortening. To such soap salt water of  $5^{\circ}$  B. is added. If changes take place after this addition, so that the soap becomes thinner, the alkali takes effect, and though no sharpness may be perceptible some palm-kernel oil, or, still better, some cocoanut-oil has to be added. This strengthens the combination and the soap recovers at once. It will stand about four per cent. of salt, and this quantity and perhaps more have to be added in order to keep the soap free from efflorescence in winter.

By using a smaller quantity of palm-kernel oil or cocoanut-oil than one-half the charge of fat, the boiling of half-grain soap is always uncertain. Soaps firm enough for cutting can be obtained by boiling with 40 per cent. of palm-kernel oil or 35 per cent. of cocoanut-oil, but hardening is effected at the expense of yield. Furthermore the soap has to be correspondingly evaporated. If this is not done tricolored soaps are formed, which though derived from the same kettle show in the frame the remarkable phenomenon of being firm and homogeneous above and below and in the centre, but dull and weak in some places and hard and piebald in others. This is due to the grain-soap not having been entirely dissolved and is corrected by again evaporating, whereby other defects will show themselves and can be remedied. Add also two to three per cent. of dissolved salt to the soap, and



should this effect the combination or fitting assist with palm-kernel or cocoanut-oil beaten up in water. Good soap shows but little sharpness and remains free from efflorescence.

The yield of such soaps when freshly cut is satisfactory, the rate depending on the evaporation. Slightly evaporated soaps lose, however, 30 per cent. by after-drying, and hence claims are frequently made on this score, though the buyer may have been satisfied with the appearance of the soap when first received. Too strong drying out is prevented by additions of water-glass and tale, but a different manner of boiling upon the combining lye becomes necessary, since the latter has to be more caustic with the use of water-glass. Silicate of soda exerts a stronger effect than salt upon easily boiling soap containing water, and hence weak lyes should never be used, as the water-glass breaks the soap up, a defect which cannot be remedied.

With the formula previously given, except that 10 to 15 per cent. of water-glass is to be used, the preparatory boiling to grain remains the same. A different lye is, however, used for the saponification of the cocoanut-oil or palm-kernel oil, for 100 pounds of oil, 50 pounds of tank-lye of  $24^{\circ}$  B., and 50 pounds of caustic soda lye of  $28^{\circ}$  B. being taken. The general custom is to add the water-glass at once to the lye, and after starting the fire to dissolve upon the boiling lye containing the water-glass the soap-cuttings and the grain. The lye becoming more concentrated by boiling, it would, however, be more advisable to first dissolve the cuttings and the grain upon the lye and then to add the water-glass and allow the whole to boil through. In introducing the palm-kernel or cocoanut-oil into the kettle, ebullition should be constantly kept up and the crutch diligently used. The addition of fat is interrupted as soon as a curly, woolly soap boiling uniformly all over the kettle is formed: 10 per cent. of the oil should be always retained for the reason previously mentioned. Test the soap as to hardness and touch, and if both be satisfactory allow it to boil quietly until no more evaporation is observed, when the fitting is regulated and salt added. Of the latter but little is, as a rule, required, especially with the use of palm-kernel oil. For palm-kernel oil one per cent. dissolved in water to  $20^{\circ}$  B. and for cocoanut-oil three per cent. can be taken

without injury. If the soap is sharp it at once boils thinner, but recovers by adding palm-kernel oil or cocoanut-oil. These soaps must be kept in constant ebullition until finished, it being difficult to raise them up again after they have once fallen down in the kettle.

The soaps may be considered finished when they uniformly break into roses up to the brim of the kettle. Fitting to touch should be very mild and scarcely perceptible. Except there be too much sharpness none of the water-glass is separated out. This defect is recognized by a sample not remaining piled up in a heap, but spreading out flat.

No one can assert that water-glass augments such soap, it being contrary to the entire manner of boiling, and, moreover, it gives a smaller yield than the preceding one, but dries out only about 20 per cent. The soap is framed hot, thoroughly and uniformly crutched, and tightly covered.

With the use of talc as a filling agent the process of boiling is the same as for the preceding soap. In working lyes of such strength as 24° and 28° B., the soap may be expected to be lacking in water and consequently boil gray and thin. But this need not trouble the soap-boiler much as long as he is sure of the correctness of the process in other respects, because the hot filling which is kept ready in a vessel standing alongside the kettle will introduce into the soap all the water and shortening wanted. The more such soap is boiled down the better it can be handled. Soap containing much water cannot be filled during the boiling, as the talc being too heavy falls to the bottom where it creates mischief.

The filling is prepared by mixing with constant stirring 100 pounds of talc with 125 pounds of hot water, and adding shortly before use two per cent. of salt. While one workman pours the filling over the soap by the ladleful, another one stirs constantly to prevent the talc from falling to the bottom. By the addition of the filling the soap should improve in appearance and boil thinner. If it is acted upon by the salt, it will look brighter but remain thin. If this be the case, add carefully palm-kernel oil or cocoanut-oil until the soap becomes thicker, and interrupt the introduction of the filling as soon as it is not immediately

absorbed by the soap, which is recognized by it falling to the bottom and scorching. The soap is finished as soon as it is saturated, it being possible to add 20 per cent. of talc in this manner. These soaps are also framed hot, crutched through, and tightly covered.

Another kind of half-grain soap, prepared according to the above formula, is augmented by crutching in the filling. The base-soap is boiled in the same manner as the preceding, the boiling to grain especially remaining the same. For combining lye half-tank lye of  $24^{\circ}$  B. and half-caustic lye of  $28^{\circ}$  B. are used. However, the filling incorporates itself with ease and regularity only in soap of little yield.

A filling agent much in use, but which, in our opinion, can scarcely be recommended, consists of two parts of crystallized soda and three parts of water-glass. The finished base-soap remains standing for two hours, when the crystallized soda is thrown into the hot soap and crutched through. When it is observed that all the crystals are dissolved, which requires but a short time, crutching is continued, and the water-glass sprinkled over the soap by means of a watering-pot and combined with it. In this manner soaps of a fair quality are sometimes obtained, but frequently they turn out faulty, the upper and lower layer being defective, and, moreover, they are inclined to efflorescence in winter. Many soap-boilers, after adding the crystallized soda and water-glass, crutch in talc and rice-flour. Stir together in as little water as possible (about 80 parts of water to 100 of flour) 1 part of talc and 2 of rice-flour and add to the mixture 20 per cent. of water-glass. This flour and talc filling is only added after the base-soap has been made "sick," as it is called, by the crystallized soda and water-glass. With correct proportions the soap is considerably augmented, the flour forming the paste which holds the mass together. The soaps have a peculiar feel when freshly cut, but improve considerably by storing. Cuttings containing flour being difficult to utilize this process is not well adapted for large factories.

Another process, which is still much in use, of preparing half-grain soap according to the above formula is as follows: The soap-cuttings are allowed to dissolve upon tank-lye of about  $22^{\circ}$



to 23° B. The cocoanut-oil or palm-kernel oil is then added and boiled to a paste-soap free from froth. The grain is next added and thoroughly boiled through. The soap is finally filled with one of the filling agents previously described. As a rule such soaps are too sharply fitted, especially when the grain has been boiled with strong lye. This is remedied by the addition of palm-kernel or cocoanut-oil.

The oldest and simplest method is, no doubt, to boil the lardaceous fats to grain with tank-lye of 12° to 13° B. The mass is boiled while in paste for a considerable time, thus insuring the saponification of all the fat, and when a cooled sample shows consistency and begins to wet when pressed between the fingers, it is salted out with 5 to 7 per cent. of common salt. The exact quantity of salt required for separation cannot be given, as it depends on the various fats. Separation with salt being effected, boiling is continued for at least one hour, and, if necessary, sufficient water added to liquefy the grain. After this the grain frequently draws together with the lye, the entire soap becoming pasty. Hence close attention has to be paid to the boiling of the grain, and samples taken with the spatula and tested whether the sub-lye is clear; otherwise boiling has to be continued until this is the case. Salt dissolved in water is then added. In case the fats are strongly colored and yield the coloring matter to the sub-lye, the latter may be drawn off or pumped out, but it is seldom necessary, as half-grain soaps are of a sufficiently light color even when the grain is boiled only upon one water. The second water is only boiled upon salt water, *i. e.*, sufficient salt is added to prevent the liquefied soap from becoming pasty. The grain-soap is covered warm and allowed to settle.

In the meanwhile a paste-soap is prepared from cocoanut or palm-kernel oil. For 100 pounds of oil, 100 pounds of tank-lye of 22° to 23° B. are brought into the kettle, and after allowing the soap-cuttings to dissolve upon it, 3 per cent. of common salt is added. Moderate ebullition is now kept up and the cocoanut-oil gradually introduced and crutched in. The whole is then allowed to boil up until any froth formed has disappeared. The use of more lye is not advisable, the quantity given sufficing in most cases. If, however, the lye is not sufficiently caustic, the

paste may become thick and pappy. In this case lye of  $22^{\circ}$  B. is added until a thin, bright paste is formed. If the first lye suffices, 250 pounds of potash solution of  $10^{\circ}$  B. are poured over the mass, and after allowing the whole to boil through, the furnace door is opened and the previously prepared grain crutched into the hot paste-soap. The addition of grain may be interrupted as soon as a thick soap is formed. The less grain is crutched in the higher the yield.

It is possible that such soap may turn out well without regulating the fitting, but it is best to be guided by samples taken as soon as the soap begins to develop net-like. If the sample be glassy but firm, salt is wanting; if glassy and soft, fitting lye. Soap remaining thin is too sharp, and thick soap remaining soft, but showing sharpness, lacks water. If obliged to add anything to soap thus crutched together, it is advisable to do it as warm as possible, and even with the furnace door open keep up sufficient fire for the bottom of the kettle to remain hot. In the worst case allow the soap to boil up, and in order to effect this with greater ease, gradually add water and fit the soap quite mildly.

If water-glass is to be used for such soaps the process of boiling remains the same, but caustic lye of  $24^{\circ}$  B. (100 pounds of lye for 100 pounds of oil) is taken for preparing the cocoanut-oil paste-soap, and after allowing the soap-cuttings to dissolve upon it, the water-glass is added. The cocoanut-oil having combined, care must be had to see that there is sufficient sharpness, otherwise the water-glass will separate out. The use of carbonated lye cannot be recommended, but about 100 pounds of salt water of  $12^{\circ}$  to  $15^{\circ}$  B. may be added to the finished paste-soap. The crutching in of the grain into such paste-soaps must be carefully executed, though actually only two defects can occur. If the soap, after adding grain, becomes glassy or viscid, lye is wanting and should be added as quickly as possible. If the soap remains thin and will not develop net-like, it is too sharp, and melted palm-kernel oil or cocoanut-oil has to be crutched in. It is always best first to make a test on a small scale. Fifty pounds of paste-soap must absorb with ease and avidity 50 pounds of grain-soap, forming a net-like mass remaining warm for some

time. The cooled sample should be white and hard and not viscid, but break off short like curd-soap.

In adding the grain to the boiling paste-soap the same directions have to be observed, fitting being regulated as much as possible before the addition of all the grain.

In the preceding it has been endeavored to describe the various methods of boiling with one and the same formula. We would remark that it is, without doubt, the best formula for half-grain soap, since, on account of the high percentage of tallow-like fats used, the resulting product may be classed among curd-soaps prepared according to the old German fashion. There are, of course, other fats, especially those of a softer consistency, which can be used, but care must be had not to take too much of them. An examination of the grain is always the best guide of how far the soap-boiler can go with such additions. There is a series of soft fats, such as cotton-seed oil, peanut-oil, sesame-oil, and also horse-fat, from which curd-soap of a tolerably firm cut, with a yield of from 146 to 150 per cent., can be boiled, but as soon as it is ground it becomes soft.

Formerly the following formula was much liked and may be recommended when good fats, tallow, and palm-kernel oil are cheap. The fats are proportioned according to their consistency, and may be replaced by others of the same consistency, for instance, bone-fat by glue-fat, cotton-seed oil by peanut-oil or horse-fat, tallow by palm-oil, etc.

Bone-fat 500 pounds, tallow 400, cotton-seed oil 100, palm-kernel oil 700.

The tallow and bone-fat are boiled to grain, but the cotton-seed oil only in case it is impure or strongly colored.

The fear that with the use of a high percentage of bone-fat or glue-fat an irregular separation of lye may take place in boiling to grain is justified. This evil may, however, be obviated by bringing first the boiling lye into the kettle and adding four to five per cent. of common salt. After heating to effect a quicker solution of the salt, the bone-fat is introduced, boiled through, and then the tallow. Should it, however, happen that the lye does not separate after adding the salt, introduce 100 pounds of palm-kernel oil and add 10 to 12 pounds of salt.



Fats boil well upon lyes from lyes of curd-soaps upon a precipitate of paste, their use serving a double purpose: the sharpness is utilized and the lye rendered clear. For the preparatory boiling to grain use tank-lye of  $13^{\circ}$  to  $14^{\circ}$  B., which is almost entirely caustic, or only pure caustic soda lye of  $12^{\circ}$  to  $13^{\circ}$  B., and boil with the addition of water until a bright, thoroughly ground-out grain is formed and the lye runs off clear. As the quality of bone-fat and glue-fat varies very much, they frequently containing dirt and water, the exact quantity of lye required for saponification cannot be given. The soap-boiler may, however, proceed in the reverse order and bring, for instance, for the 900 pounds of fat, 1800 pounds of lye of  $12^{\circ}$  to  $13^{\circ}$  B. into the kettle, and when he finds non-utilized sharpness to be present add fat which will readily incorporate itself. The content of dirt and water can then at the same time be approximately calculated.

Though the formula calls only for 20 per cent. more of combining fat than the preceding one, a different combining lye is nevertheless required in order to effect the saponification of the palm-kernel oil, which belongs to the fats which can only be saponified with pure caustic lyes. Take 500 pounds of tank-lye of  $24^{\circ}$  B. and 200 pounds of pure caustic lye of  $25^{\circ}$  B., dissolve upon it the soap-cuttings, add the grain, and boil until a clear grain free from froth lies in the kettle. To many a one this may seem a singular requirement, but there are good reasons for it. Under different conditions grain-soaps boiled from fats give varying results, one of the injurious ones being that frequently a defective separation of the sub-lye in a hot state takes place. By adding such wet grain to the combining lye in the kettle, much saline lye is introduced, the result being a saline augmentation which subsequently is frequently fatal to success. However, by evaporation the lyes, though saline, are sufficiently concentrated to enter into correct saponification with the palm-kernel oil. The content of salt does no harm, it even belongs to every half-grain soap, but care must be had not to add subsequently carbonated lye or salt. It is still better and more rational to have always a supply of cold grain on hand, as it not only facilitates the entire work, but makes it possible to boil at any moment half-grain soap in one kettle, whereas, if the grain is boiled on the

same day, two kettles are required or the grain has to be ladled out. Discharging or pumping out the sub-lye does not suffice, because all such fats as bone-fat or glue-fat deposit a strong slime, which adheres so tightly to the bottom of the kettle that it can only be removed by scraping.

Keeping grain on hand is so convenient that it will not be given up when once introduced. The process of preparation is as follows: All sub-lyes, or lyes derived from soaps upon a precipitate of paste and containing no resin, are left in the kettle, and 1000 to 2000 pounds of fat, according to what may be required, added. As soon as the mass boils, caustic soda lye of  $14^{\circ}$  to  $15^{\circ}$  B. is introduced until it is noticed by the grain-soap showing pressure that all the fat is saponified. As a rule these grain-soaps do not boil to paste, the lyes from soaps upon a precipitate of paste containing sufficient salt to prevent it. But even without such lyes the conversion of the fats into such soap is of advantage, since it prevents loss by leakage of barrels, etc.

The finished grain is best brought into small, low sheet-iron boxes, about 5 feet long,  $2\frac{1}{2}$  feet wide, and  $5\frac{3}{4}$  inches high, in which it becomes cold overnight. It is then cut into slabs, which are stored away, each kind of grain by itself, so as not to create confusion when yields are to be calculated.

The cold grain is brought together with the soap-cuttings upon the combining lye, and as it dissolves as quickly as the latter there is no loss of time. Another advantage is that each day a boiling of half-grain soap can be finished in one and the same kettle, which cannot be well attained in any other manner.

By using such cold grain one is sure of the lye adhering to warm soap having settled. About two per cent. of salt, if not already added to the combining lye, is then introduced, next the cotton-seed oil, together with 100 pounds of lye of  $24^{\circ}$  B. required for its saponification, and the whole boiled thoroughly. In this manner the cotton-seed oil is best decolorized and rendered inodorous, and also loses its property of later on producing spotted soaps. Finally the palm-kernel oil is introduced, and allowed to combine with moderate ebullition and assistance with the crutch. When the soap boils high and uniform, it is tested as to fit.

All superfluous, premature doctoring, especially with so-called shortening agents, should be let alone. If the test shows the soap too sharp, add carefully palm-kernel oil previously stirred in water (100 pounds of oil in 100 pounds of hot water); if it is viscid and dull, sprinkle caustic soda lye of 25° B. in small portions over it. It may further happen that, though the soap boils high and bright, a cooled sample is not white but colored. Such soap requires shortening, which, in the winter months, has to be effected with salt. Dissolve 10 pounds of salt in 20 pounds of hot water, and gradually add the solution until the soap breaks into white curly roses all over the kettle and "dies off" white. As a rule such soap shows sharpness after adding the salt water. Should this be the case, introduce more palm-kernel oil stirred in water. For the summer months soda or potash solution may be used besides salt, whereby the yield is increased and rapid drying out decreased.

This soap being boiled without filling, strong evaporation is not required; hence, if too much water has been lost by evaporation, it is best replaced by diluting the salt solution. These soaps, which are thinly fluid as compared with those filled with water-glass, work very quickly in the frames, and hence, if allowed to stand hot and covered, defective and distorted flux would be formed. They are therefore several times crutched in the frames and tightly covered. Such soaps are decidedly the best as regards appearance, are salable as soon as cut, and give a greater yield than can be obtained from the formula by boiling in any other manner.

Cocoanut-oil gives more solid soaps and forms a stronger combination than palm-kernel oil, also transferring these properties to soaps in the preparation of which it is only partially used or as a combining fat. In half-grain soaps the particles of soap must possess a certain mobility, so that the formation of flux may take place. This is effected by the addition of salt solutions, about 12 per cent. of salt being required when the proportion of cocoanut-oil to other fats is as 7 : 10. By the addition of such large percentage of salt the resulting soap is, however, rendered rough and hard, which is modified by the use of fats of softer consistency.



By boiling, for instance, 600 pounds of bone-fat or glue-fat, 200 pounds of cotton-seed oil, 200 pounds of horse-fat, and 700 pounds of cocoanut-oil, soap with a firm cut can be obtained.

The boiling to grain is effected in the same manner as previously described, but a different combining lye has to be used, half tank-lye of  $24^{\circ}$  B. and half caustic soda lye of  $28^{\circ}$  B. being employed. If the cotton-seed oil is not boiled to grain, the same lye is used for its saponification. For the formula given 450 pounds of tank-lye of  $24^{\circ}$  B. and the same quantity of caustic soda lye of  $25^{\circ}$  B. are required. The lye is brought into the kettle together with 5 per cent. of the cocoanut oil of salt (in this case 35 pounds), and after dissolving the grain and soap-cuttings upon it, the larger portion of the cocoanut-oil is introduced with moderate boiling, and the establishment of combination assisted with the crutch. Of the 700 pounds of cocoanut-oil prescribed in the formula, at least 50 pounds are reserved for subsequent correction in fitting. Such soaps, as a rule, rise beautiful and bright, and during ebullition show all the properties of a well-boiling half-grain soap. Salt water should, however, be always kept ready and added in case the formation of a more viscid combination is observed. As above mentioned, such soaps will stand from 10 to 12 per cent. of salt, and it may be added until it is observed to produce an effect by the soap boiling lighter and thinner. If an excess of sharpness is noticed, try to fix it by the addition of the reserved cocoanut-oil, and if this does not suffice add more oil mixed with hot water until the soap boils light and a sample cools slowly. No more carbonated lye than that contained in the combining lye is used in winter, though in summer the soaps will stand more of it, calcined soda being frequently scattered over the boiling soap for shortening. Such radical means should, however, be only used when absolutely necessary and water is to be withdrawn from a weak, thin soap.

By using the same proportions of fat (10 : 7), with the exception that a portion of the cocoanut-oil is replaced by palm-kernel oil—say 400 pounds of palm-kernel oil and 300 pounds of cocoanut oil—the other fats must be taken into consideration if the soap is to be firm in cut and salable.

The following formula may serve for the purpose: Bone-fat

600 pounds, lardaceous fats 200, horse-fat or cotton-seed oil 200, palm-kernel oil 400, cocoanut-oil 300.

The process of boiling to grain is the same as for the preceding formulas, the same combining lye being also used, or it might be modified in so far as to take 500 pounds of tank-lye of  $24^{\circ}$  B. and 400 pounds of caustic soda lye of  $24^{\circ}$  B. The soap-cuttings and grain are dissolved upon the lye, the cotton-seed oil being boiled at the same time. If the cocoanut-oil is first brought into the kettle, 5 per cent. of salt may be safely added to the lye. As in the preceding process, 50 pounds of palm-kernel oil are reserved and the rest of the operation carried out in the same manner. By sufficiently shortening the soap with salt it is not sharp and remains free from efflorescence in the severest winter, but it must be crutched in the frame. Frequently it lies heavy in the frame, as if cooled too much. This, however, is only deceptive, the soap when covered becoming again heated. It is crutched until it shows a temperature of  $171.5^{\circ}$  F., at which it forms best.

For the manufacture of half-grain soap according to the following formula: Lardaceous fats 400 pounds, bone-fat 600, and palm-kernel oil 700, with about 15 per cent. of water-glass filling, the lardaceous fats and bone-fat are first boiled to grain upon caustic lye of  $13^{\circ}$  to  $14^{\circ}$  B. The combining lye for the palm-kernel oil consists of 200 pounds of tank-lye of  $24^{\circ}$  B. and 500 pounds of caustic soda lye of  $25^{\circ}$  B. After dissolving the soap-cuttings and the grain upon the lye, about 100 pounds of water-glass are added and allowed to boil through. Moderate ebullition being kept up, the palm-kernel oil is then introduced, reserving, however, 100 pounds of it. The soap being uniformly boiled through, it is tested whether it has sufficient lye-power to absorb the rest of the palm-kernel oil. If such be not the case, about 2 per cent. of salt (in this case 14 pounds) dissolved in water is added in portions, but never at once. This being boiled through and still no lye-sharpness observed, about 50 pounds of caustic soda lye of  $25^{\circ}$  B. are added and then the palm-kernel oil. As a rule, half-grain soaps show perceptible sharpness after the addition of salt, a proof that the salt commences to act, and hence great care has to be exercised in the

further addition of salt solution. All the palm-kernel oil being in the kettle, and the soap boiling high and light, the fit is so regulated as to shorten either with salt water or carefully using caustic lye of  $24^{\circ}$  B.

Only in rare cases can solutions of the carbonates of the alkalis be added to such soaps boiled with water-glass, and to judge of the necessity of such addition requires experience. The soap must boil light and break into roses over the entire kettle. The samples must not spread out flat, but when cold still show the rings of the heaping.

Half-grain soaps boiled from 1000 pounds of good bone-fat and 700 pounds of palm-kernel oil are also filled with talc.

The grain is boiled upon lye of  $13^{\circ}$  to  $14^{\circ}$  B., to which about eight per cent. of salt (in this case 80 pounds) is at once added, by which the formation of a paste-soap is prevented, which later on could not be separated by means of salt. Such grain boiled from bone-fat alone, is generally bright and an addition of water is seldom required.

The combining lye for the palm-kernel oil consists of 500 pounds of tank-lye of  $24^{\circ}$  B. and 200 pounds of caustic soda lye of  $25^{\circ}$  B. If soap-cuttings containing talc are on hand, it is advisable first to bring the lye to ebullition, the kettle being covered, and then dissolve the cuttings upon it, this preventing the talc from settling solidly on the bottom of the kettle. The palm-kernel oil is then added, assisting with the crutch and boiling in as thickly as possible. Stir the talc in hot water to a homogeneous paste and shortly before using it add two per cent. of salt : 100 pounds of talc, 100 pounds of hot water, and 4 pounds of salt dissolved in 20 pounds of hot water constitute the filling. Pour some of the solution over the boiling soap and crutch in so as to effect a uniform distribution as quickly as possible. To a healthy and lightly boiling half-grain soap the filling can only be added with great care, it being necessary to allow the water adhering to the talc to evaporate before introducing a fresh portion of the filling, as otherwise the talc falls down and burns fast to the bottom of the kettle, which causes hard dots in the soap. The filling incorporates itself with greater ease in an over-evaporated soap, and hence soap lacking water can be improved by it,



the adding of it being interrupted as soon as the soap boils light and high. The fit has now to be tested. If the addition of salt affects the combination, pour melted palm-kernel oil over the soap, and if it becomes thicker more of the filling may be introduced. It is impossible to state the quantity of filling half-grain soap will take up, it depending entirely on the quantity of salt-solution it will absorb, because the talc itself is an entirely indifferent body towards the soap.

From a combination, in which the paste-forming fats are to the others as 7 : 10, a soap firm in cut can also be obtained with 15 per cent. of water-glass.

By taking, for instance, 500 pounds of bone-fat, glue-fat, or lard, 200 pounds of cotton-seed oil, sesame-oil, or peanut-oil, 300 pounds of tallow or palm-oil, 400 pounds of palm-kernel oil, and 300 pounds of cocoanut-oil, the 500 pounds of bone-fat and 300 pounds of tallow alone are boiled to grain, while the cotton-seed oil is brought into the kettle together with the combining lye. If, however, the cotton-seed oil is replaced by sesame-oil or peanut-oil, the latter can be boiled to grain together with the bone-fat and tallow.

For combining lye for 400 pounds of palm-kernel oil, 300 pounds of cocoanut-oil, and 200 pounds of cotton-seed oil, about 900 pounds of caustic soda lye of 25° B. are required. The soap-cuttings having been dissolved upon the lye, the cotton-seed oil and the grain are introduced. When the whole is thoroughly boiled through 100 to 130 pounds of water-glass are added, and after again allowing the mass to boil up scatter 25 pounds of salt over it, and then add first the cocoanut-oil and next 300 pounds of palm-kernel oil, reserving the remaining 100 pounds for future use. When the soap boils up thick and curly, it is tested as to whether it contains sufficient sharpness to allow of the introduction of the reserved 100 pounds of palm-kernel oil, and if such be the case it is added in portions. If on testing the soap the samples are found to become glassy, salt is wanting and has to be added in the form of a solution of 20° B. until the soap loses this property. In doing this sharpness will, however, appear, which is neutralized by the addition of palm-kernel oil. The fit is regulated only after the entire quantity of reserved

palm-kernel oil is in the kettle. Such soap must boil high and light, otherwise water is wanting, the addition of which is readily accomplished if weak salt solutions can be used. But if a sample cools off like grain-soap, no more salt solution can, as a rule, be introduced, but the soap will take up water, of which, however, no more than is absolutely necessary must be added. If the water-glass remains unaltered in the soap, sufficient lye is present even if sharpness is not plainly observed. Hardening is effected by salts and light boiling by the addition of water. In case the samples do not heap up in a pile but spread out flat, palm-kernel oil has to be added, though frequently only a small quantity is required. The finished healthy soap boils uniformly over the entire kettle and breaks into roses up to the rim.

If tale is to be used for filling and a portion of the palm-kernel oil to be replaced by cocoanut-oil, fats of a softer consistency than bone-fat can be employed, tale filling and tallowy fats giving, in the presence of cocoanut-oil, rough and brittle soaps. The following may serve as a formula: Bone-fat 600 pounds, horsefat 200, cotton-seed oil 200, cocoanut-oil 400, palm-kernel oil 300.

The 600 pounds of bone-fat and 200 pounds of horse-fat are boiled to grain upon tank-lye of  $13^{\circ}$  B., to which four to five per cent. of salt has been added, the result being a light soap from which the lye subsides well. After-grinding with water is seldom required.

The combining lye for the above formula consists of 500 pounds of caustic soda lye of  $25^{\circ}$  B. and 400 pounds of tank-lye of  $24^{\circ}$  B. The grain, together with the 200 pounds of cotton-seed oil, is first thoroughly boiled upon the lye, the soap-cuttings, if containing tale, are then added, next the cocoanut-oil, and finally 200 pounds of palm-kernel oil, 100 pounds of it being reserved for correction. Boiling is now continued, and in order to introduce salt into the soap, which in any case is too sharp, add the reserved palm-kernel oil and some of the tale liquor. After adding the latter, the soap must improve in appearance and especially boil thicker, otherwise it is too sharp. The principal point, however, is to keep the soap in constant ebullition, crutching and stirring it and putting off the regulating as long as pos-

sible, until at least 20 per cent. of the filling (in this case about 140 to 150 pounds) is in the kettle. By now pouring melted palm-kernel oil over the soap, by which it is improved, 10 per cent. more of the filling can be added and then the fit regulated. If it is found that water is wanting, the talc liquor can be diluted to double its volume and added in that state. The finished soap must boil thick and break into roses over the entire kettle. The samples should heap up, break like grain-soap, and be entirely white. After resting for one hour in the kettle, the soap should show mottling. It is framed hot, crutched through, and covered when cooled to about 185° F.

The formulas for half-grain soaps most used consist of half combining fats and half other fats, provided there be no material difference in the price of the fats. The soaps are filled with carbonated lye and salt, the yield being about 200 per cent., or under the most favorable conditions, with the use of palm-kernel oil or tallow, 208 per cent.

The following formula yields a soap firm in cut: Bone-fat 700 pounds, cotton-seed oil 100, horse-fat 200, palm-kernel oil 1000.

The bone-fat or horse-fat or other fats of similar consistency are boiled to grain with tank-lye of 13° to 14° B., or caustic soda lye of 12° to 13° B. The entire quantity of lye (in this case about 1800 pounds) being brought into the kettle, the fire is started or steam admitted. With the use of direct steam, the water of condensation passes into the kettle, diluting the lye. Hence its strength must be increased 1½° to 2° and a correspondingly smaller quantity taken. After adding 5 per cent. of salt to the lye, the fat is introduced, which, by assisting with the crutch, generally saponifies before the mass reaches ebullition. The whole is then allowed to boil through and the wanting salt added. If the grain formed is round and bright, nothing further is done with it, since, with the kettle tightly covered, the sub-lye subsides sufficiently in 10 to 12 hours. It is, however, recommended to ladle the grain into small boxes and allow it to cool.

As combining lye for 1000 pounds of palm-kernel oil and 100 pounds of cotton-seed oil, 900 pounds of tank-lye of 24° B., and 200 pounds of caustic soda lye of 24° B. are used. Dissolve the soap-cuttings upon the lye, and after boiling the 100 pounds of



cotton-seed oil through with it, add the grain. Now evaporate until a thick grain is formed, then add 900 pounds of the palm-kernel oil, and after having effected a good combination by slow boiling, test the fit. It may happen that a healthy soap, correctly boiling in all proportions, is already in the kettle, which would be due to the carbonates which tank-lye of  $24^{\circ}$  B. may contain. For the summer months such soap might suffice, but would be unfit for winter, as it would show much efflorescence. By the addition of salt such soap can, however, be improved and rendered free from efflorescence without the further introduction of lye. The salt helps to augment the soap, and liberates sufficient lye completely to saponify the reserved 100 pounds of palm-kernel oil. By using the salt, not direct, but as a strong solution, the process is not difficult and is entirely free from danger.

By adding to such soap, apparently boiling correctly, only the 100 pounds of palm-kernel oil, at least the same quantity of water or lye would have to be introduced to keep up the right condition of boiling. However, by adding, instead of water or lye, an equal quantity of salt solution, it will be found that the condition of boiling undergoes no change. For the above formula, salt solution of  $10^{\circ}$  B. will, as a rule, suffice, and as water evaporates by boiling, about 120 pounds of salt solution of  $10^{\circ}$  B. can be, without injury, used for the 100 pounds of palm-kernel oil. By again testing the fit, especially in regard to the hardness of the soap, complete regulation is very simple. Such soaps should be bright, and while breaking into roses over the entire kettle, not boil thick.

In the preceding pages various processes of boiling have been given which are used for the manufacture of half-grain soap from approximately similar fats. While the principal object of the preparatory boiling to grain is to purify and render less odorous impure, badly-smelling, or colored fats, it has also been shown that fats of a tallowy consistency, such as palm-oil, and fats of a lardaceous consistency, such as horse-fat and bone-fat, can also in this manner be worked into half-grain soap with greater ease and profit. An entire series of fats—in fact, nearly all of them—can first be boiled to grain and then used for half-grain soap, but a

better and surer result and greater yield is obtained from them by direct boiling.

A smaller proportion of fats than 7 parts to 10 parts of combining fat may also be used for indirect boiling, but in this case the process approaches that of direct boiling, as the small percentage of grain becomes of little importance. We would further mention that impure sedimentary oils containing much stearin—from cotton-seed oil, rape-oil, poppy-oil, sesame-oil, and the sediment obtained by bleaching linseed-oil with lye—cannot be better utilized than for half-grain soap. For the sake of cheapness, dark oleic acid and cotton-seed oil can also be worked for half-grain soap, but not more than 50 per cent. of them should be used, even with cocoanut-oil as combining fat. In order to obtain soaps of a firm cut, 40 pounds of cotton-seed oil, oleic acid, etc., may be used for 100 pounds of cocoanut-oil, but only 30 to 35 pounds of linseed-oil. All these oils are boiled upon strong, generally saline, lyes of  $20^{\circ}$  B., and sedimentary oils containing much dirt and water even upon caustic lyes of  $25^{\circ}$  B. The process of boiling is the same for all. The lye is made boiling hot and the oil crutched in. When saponification has apparently taken place, *i. e.*, when no more clear oil floats upon the lye, the mass is boiled through for the particles of dirt to separate and settle as slime upon the bottom of the kettle. It cannot be recommended to use such grain warm, it being best to ladle it into barrels, cover them, and later on draw off the sub-lye from the cold soap.

*Half-grain soap by the direct method.*—The principal and frequent defect of half-grain soap is that the ground is not of one color and the mottling does not stand out prominently upon it. A white ground being preferred, a gray or yellowish shade is rendered especially conspicuous with a bright red or blue marbling. In a gray mottled soap a moderately colored ground is frequently quite handsome and makes the soap resemble ordinary full household soaps. Generally speaking, it is, however, absolutely necessary to work the fats pure and free from dirt and not too strongly colored, since the particles of color and dirt pass later on into the marbling and destroy the bright and fresh appearance of the color. Impure fats must, therefore, be melted and allowed to

settle, and colored fats bleached. Direct boiling being safe and remunerative only with entirely pure fats, those contaminated with acidulated water must be especially rejected.

Butchers frequently render the crude tallow with an enormous quantity of sulphuric or hydrochloric acid, and in ladling the melted tallow from the acid water do not particularly care if some of it goes into the barrel together with the tallow. With such material it is next to impossible to prepare faultless half-grain soap by the direct method, especially if water-glass is to be used.

As in boiling by the direct method nothing is separated, everything passing into the soap, more attention has to be paid to the lyes than in boiling by the indirect process, and hence this method can only be recommended for factories provided with reservoirs for the previous clarification of the lyes, and besides, with caustic soda lyes of any desired strength. Directly boiled half-grain soaps are much more sensitive than those boiled upon grain, because the salt frequently comes much earlier into use. Recourse to the direct process is, however, absolutely necessary where linseed-oil, cotton-seed oil, or oleic acid is to be worked.

The lyes used are similar to those for indirect boiling, consisting of tank-lyes of  $20^{\circ}$  to  $30^{\circ}$  B., or lyes of the same strength prepared from caustic soda and 30 per cent. calcined soda. Moreover, caustic soda lyes of  $30^{\circ}$  B., and even of  $40^{\circ}$  B., must be kept ready in case of need, as well as salt solution of  $24^{\circ}$  B. and soda or potash solution of  $30^{\circ}$  B. If the preparation of these solutions were left to the time when actually needed, their application would, as a rule, be too late.

In the choice of the combining lyes all the fats worked have to be taken into consideration, it being a principal condition that with the use of cocoanut-oil, palm-kernel oil, linseed-oil, cotton-seed oil, peanut-oil, etc., all of which only saponify with caustic lyes, the boiling is first executed upon caustic lyes as pure as possible, and the required carbonated lye only used later on.

We submit the following formula: Lardaceous fats or palm-oil 500 pounds, horse-fat or cotton seed oil 200, bone-fat 200, oleic acid 100, cocoanut-oil 350.

For this formula an average lye of about  $24^{\circ}$  to  $25^{\circ}$  B. is required—namely, about 800 pounds of tank-lye of  $23^{\circ}$  B., or of



lye of the same strength prepared from caustic soda lye, with an addition of 30 per cent. calcined soda, and 600 pounds of caustic soda lye of  $25^{\circ}$  to  $30^{\circ}$  B. The boiling is generally executed by first bringing the weaker tank-lye into the kettle and allowing the soap-cuttings to dissolve upon it. If any of the fats, such as oleic acid, are to be decolorized or to a certain extent subjected to a preparatory boiling, 200 pounds of caustic soda lye are at once added and the linseed-oil, cotton-seed oil, or oleic acid allowed to boil through with it. The horse-fat and lardaceous fats are then introduced, care being had that a quantity of lye corresponding to the quantity of fat (100 pounds of lye to 100 pounds of fat) boils in the kettle, this facilitating the formation of a correct judgment in case of trouble. If a combination is not formed, add 100 pounds of cocoanut-oil together with 100 pounds of caustic soda lye, and wait, keeping up quiet ebullition, until combination is established. Then add the remaining fats, with the necessary percentage of lye and also the cocoanut-oil, reserving, however, about 100 pounds of it. Even without these 100 pounds of cocoanut-oil, a soap breaking into roses and having a bright, though weak, appearance, must now boil in the kettle. Then add about 2 per cent. of salt, which makes the soap more beautiful and facilitates its development. Should, however, the soap, after the addition of the salt, boil thinner or even "wet," add the cocoanut oil together with an equal quantity of water. If a thick, curly soap now boils in the kettle, ascertain by a sample whether sufficient lye is present. With the use of not more than the prescribed quantities of lye nothing can be wanting in a soap boiling faulty except water lost by evaporation, sufficient of which is added until the soap, which should be kept in constant ebullition, boils high and bright. It must, however, not paste, but always boil in laminae and also break into roses. The fit has also to be frequently tested, because the more water the soap absorbs the more it can be augmented, the augmentation consisting of slightly caustic, saline lye of about  $8^{\circ}$  B. As a rule, the tank-lye conveys as much carbonate into the soap as required, but in summer it may be necessary to use more, which is recognized by the soap being well-combined and bright.

If too much water has been introduced in augmenting with lye

so that the soap boils in laminæ, it is evaporated until it breaks again into roses, when it may be considered finished. Strong evaporation is not necessary, this soap when fit for cutting presenting a better appearance than that with large marble which is strongly evaporated. Marbling, if desired, forms as well in soaps containing water.

A formula similar to the preceding, except that palm-kernel oil instead of cocoanut-oil is used as combining fat, consists of: Tallow or palm-oil 600 pounds, bone-fat or horse-fat 300, cottonseed or linseed-oil 100, palm-kernel oil 450.

The boiling lye, which is the same for all the fats, consists of 800 pounds of tank-lye of  $23^{\circ}$  B. and 600 pounds of caustic soda lye of  $25^{\circ}$  to  $30^{\circ}$  B., *i. e.*, an average lye of  $25^{\circ}$  B.

Dissolve the soap-cuttings, and at the same time boil the cottonseed or linseed-oil through upon the tank-lye of  $23^{\circ}$  B. and then add the tallow. If combination is not formed, introduce cocoanut-oil together with the required lye. To prevent disturbances care should be had that 100 pounds of lye are always in the kettle for every 100 pounds of fat. When everything is in the kettle except 100 pounds of palm-kernel oil and 100 pounds of caustic soda lye of  $30^{\circ}$  B., try to prepare a soap by boiling according to the half-grain method. Add gradually 20 pounds of salt dissolved in water and also 20 pounds of crystallized soda, the effect of which must be ascertained by samples. In case they are glassy and show no lye-sharpness, add caustic lye until they become soft. By continued boiling too much water may have been evaporated, and, therefore, add gradually 100 pounds of it, and if the soap improves thereby, still more until it is bright and boils high and in laminæ. The soap should now boil faultlessly; otherwise salt or carbonated lye may be wanting, and, in this case, potash solution has to be added. The reserved 100 pounds of palm-kernel oil are now introduced and fitted with about 70 pounds of caustic soda lye of  $30^{\circ}$  B. In case the entire soap does not close together and the samples are glassy, salt water has to be added until the samples become again soft, but first let the soap boil up until it is thick and breaks into roses over the entire kettle. The soap need not be evaporated thick,

but is crutched in the frames and covered when cooled to  $167^{\circ}$  F.

The following may serve as a formula for a soap to be filled with water-glass and cocoanut-oil as combining fat: Tallow or palm-oil 500 pounds, bone-fat or horse-fat 300, peanut-oil 200, cocoanut-oil 500.

The boiling lye for all the fats consists of 600 pounds of tank-lye of  $23^{\circ}$  B. and 900 pounds of caustic soda lye of  $25^{\circ}$  B. About 150 pounds of water-glass are used. The soap-cuttings having been dissolved upon the 600 pounds of tank-lye and 400 pounds of caustic soda lye, the tallow, peanut-oil, and bone-fat are introduced, and if combination is not established add 100 pounds of cocoanut-oil. When combination is formed gradually introduce the caustic lye in which the 150 pounds of water-glass have been previously dissolved. The soap, of course, tears apart and forms a thick grain. When all is thoroughly boiled through the melted cocoanut-oil is gradually added, and combination being re-established samples are constantly taken which will clearly indicate any excess of lye. The soap now boils thick and curly, and if the samples "die off" glassy salt is wanting. To rough, dull-appearing soap potash solution is first added, but to bright soap salt water of  $22^{\circ}$  B. Sprinkle a small quantity of it over the soap, but interrupt the adding immediately on its taking effect. As a rule lye is liberated which is fixed by the addition of oil. If after this the samples remain soft, the remaining cocoanut-oil, together with the required lye, is added. Soaps containing water-glass should be kept thicker and must uniformly break into bunches of roses over the entire kettle. Samples of the soap when boiling up finished should be white and soft, otherwise salt water must be added until this is the case, and generally some oil has also to be introduced. The soap is framed hot and crutched through.

The direct method of boiling half-grain soap is of more importance when for 100 parts of cocoanut or palm-kernel oil about 30 parts of light oil, such as cotton-seed, linseed, sesame-oils, oleic acid, lard-oil, etc., are to be used. During the dull times in Germany a few years ago some soap-boilers even used as much as 50 parts of these oils to 100 parts of cocoanut or palm-



kernel oil, but the production of salable soap from such a combination requires great experience.

By taking, for instance, the following formula, which is much used, palm-kernel oil 750 pounds and linseed-oil 250 pounds, it must be at once explained that, though a firm soap can be obtained by direct boiling, the yield, without filling, will only be 185 to 190 per cent. And notwithstanding that only caustic soda lye with a few per cent. of salt and a small quantity of carbonated lye can be used for boiling, the soaps are very thinly fluid and must be for some time crutched in the frames to prevent the formation of distorted flux.

With these soaps it would seem as if water-glass and talc actually produced augmentation.

The boiling lye consists of 1000 pounds of caustic soda lye of 25° B., to which 30 pounds of salt can be at once added. Bring about 800 pounds of the lye into the kettle, dissolve the soap-cuttings upon it, and at the same time boil the linseed-oil through with it, whereby it will be decolorized. Now add 500 pounds of palm-kernel oil, and assist with the crutch in order to bring about combination as soon as possible. The soap rises in the kettle, and is kept high and tested whether it has sufficient touch. If the soap is not to be augmented with water-glass, or talc, about six to eight per cent. of crystallized soda is now added and then alternately palm-kernel oil and fitting lye.

To fill such soap with water-glass the process is the same, only instead of crystallized soda, 15 per cent. of water-glass, mixed with 100 pounds of caustic soda lye of 25° B., is added to the soap. The whole being thoroughly boiled through, palm-kernel oil and fitting lye are alternately introduced. A larger quantity of salt than that given can, as a rule, not be used, but 10 to 20 pounds of crystallized soda may still be required. Salt acts quickly and strongly upon such soaps boiled with linseed oil, and hence it is advisable to be careful with it. The soap boils light and bright, but lies very thinly fluid in the kettle and in the frame. By crutching it repeatedly the mottling will stand.

By replacing the linseed-oil by cotton-seed oil, the preceding method of boiling can be modified by treating a charge of 750

pounds of palm-kernel or cocoanut-oil and 250 pounds of cotton-seed oil in the following manner :—

Use as boiling lye 500 pounds of caustic lye of  $20^{\circ}$  B. and the same quantity of  $30^{\circ}$  B., which corresponds to an average lye of  $25^{\circ}$  B. Bring first the lye of  $20^{\circ}$  B. into the kettle and add the soap-cuttings and the cotton-seed oil. When the lye is warm endeavor to crutch in the cotton-seed oil, it combining best with lye of  $20^{\circ}$  B. at  $144.5^{\circ}$  F. Now introduce about 200 pounds of palm-kernel oil, so that a complete paste-soap is formed, the operator being then sure that all parts of the cotton-seed oil have entered saponification and that flocculent soap will not be formed later on, and also that the finished product will keep white when stored. The soap is then separated with the lye of  $30^{\circ}$  B., any water-glass to be used being added at the same time.

By this method of boiling, a preparatory boiling to grain has to a certain extent been effected without the extra labor.

Now add, when palm-kernel oil is used, 4 per cent. = 40 pounds, and when cocoanut-oil is used 5 per cent. = 50 pounds of salt ; allow the whole to boil thoroughly through and then gradually add the palm-kernel oil. By carefully introducing the oil in a liquid state the presence of water-glass will not be injurious. Before adding the last portion of the oil, which to a certain extent serves for fitting, allow the whole to boil up strongly. This soap may sometimes require carbonates, and either crystallized soda or potash solution is added. To soap evaporated too thickly, which rises heavy in the kettle, add water, or in case this is not absorbed, weak salt water. As a rule, some fat is also wanting. The finished soap should be thick and break uniformly into roses over the entire kettle up to the rim. Samples must be soft and remain heaped up in a pile, otherwise the soap is too sharp.

Peanut-oil is used in larger quantities than the preceding oils (linseed-oil and cotton-seed oil). By taking 40 pounds of it to 100 pounds of palm-kernel oil and 50 pounds to 100 pounds of cocoanut-oil, boiling is readily executed.

A charge of 1000 pounds of palm-kernel oil and 400 pounds of peanut-oil can be boiled with 400 pounds of tank-lye of  $23^{\circ}$  B. and 1000 pounds of caustic soda lye of  $25^{\circ}$  B. Bring the

400 pounds of tank-lye and 600 pounds of the caustic soda lye into the kettle, dissolve the soap-cuttings upon it and then add the peanut-oil and 600 pounds of palm-kernel oil. Combination being established, introduce the water-glass mixed with caustic lye and at the same time 3 per cent. of salt for palm-kernel oil and 4 per cent. for cocoanut-oil, and allow the whole to boil thoroughly. Remove any sharpness present by the addition of palm-kernel oil and fit mildly. Though, as a rule, sufficient carbonates are introduced by the tank-lye, should any be wanting add potash solution, which makes the soap more delicate and lustrous.

Oleic acid is also used for direct boiling, but as it varies very much in quality, it is necessary accurately to know how much to use, though the quantity should never exceed 30 per cent. It is also advisable to use, besides oleic acid, a fat of tallowy consistency or good bone-fat, for instance, 1000 pounds of palm-kernel oil, 300 pounds of oleic acid, 200 pounds of tallow, etc. About 700 pounds of tank-lye of  $23^{\circ}$  B. and 700 pounds of caustic soda lye of  $27^{\circ}$  to  $28^{\circ}$  B. will have to be used. Bring the 700 pounds of tank-lye and 400 pounds of caustic soda lye, together with the soap-cuttings, into the kettle, and the latter being dissolved, add the oleic acid in portions, using the crutch vigorously to prevent the formation of lumps, and only after it boils thoroughly introduce the lardaceous fats. Then add sufficient palm-kernel oil to fix the sharpness, and when the soap boils thick introduce 3 per cent. of salt, and later on the remaining caustic lye mixed with water-glass. Such soap can also be filled with talc, which removes the gray or brown tint that oleic acid frequently imparts to it.

Grain-soaps boiled with oleic acid are inclined to subside when there is an excess of sharpness or much salt has been used, the water-glass or talc being also affected. Care must, therefore, be had that before all the lye is in the kettle the soap is not too sharp and boils faultlessly, and that ebullition is not interrupted until everything has been introduced. The finished soap generally lies heavy, very close, and dark in the kettle. Generally speaking, oleic acid is not a very suitable fat for half-grain soap.

*Half-grain soaps from base-soap.*—The preparation of these soaps, which is to some extent carried on in England and the



United States, varies but little from that of augmented grain-soaps, and experience in the fabrication of the latter is required before it is advisable to undertake the manufacture of this kind of half-grain soap. The principal condition is that the base-soap be correctly boiled, otherwise augmentation cannot be effected. Caustic soda lye, to be sure, facilitates boiling and makes it more certain, but the fats used must always be taken into consideration. The formulas vary as much as those for boiling half-grain soap by the direct and indirect methods. Augmentation is effected with talc and crystallized soda, though chiefly with water-glass. A charge which can be handled with comparative ease consists, for instance, of bleached palm-oil 500 pounds, lardaceous fats 500, palm-kernel oil 500.

The 1500 pounds of fat are boiled to grain with caustic soda lye of  $15^{\circ}$  B., which is, of course, effected with the greatest ease and safety by means of steam. The preparatory boiling in paste requires the greatest attention, for if the saponification of the fats is defective, the resulting grain either accepts no augmentation or breaks up. The fats which do not form a firm combination, in this case the palm-oil and tallow, are first brought into the kettle and boiled until a paste-soap showing pressure is formed. The palm-kernel oil and the remaining lye are then added and boiling is continued until the soap begins to "wet." Now sprinkle eight per cent. of salt dissolved in hot water over it and allow the whole to boil up. With the use of steam the soap boils at once clear. Boiling is continued for two hours. With an open fire it may happen that by the evaporation of water a thick clear grain is formed. If such should however not be the case, boiling is interrupted for a short time to give the salt-lye a chance to subside, which is then drawn off or pumped out, allowing only so much to remain under the soap that the latter can form a clear, strong grain upon scant lye. As long as this is not the case boiling cannot be finished. The bubbling grain is then ground by sprinkling water over it until it is liquefied and boils in laminae. The sub-lye may be thick, but must not form paste. Now cover the kettle for 24 hours, then skim off the froth, and remove the sub-lye entirely if possible.

To add the filling, crutching or mixing machines, which have

already been described, are necessary for working on a large scale. A test with about 50 pounds of the base-soap is, however, first made to ascertain what proportion of water-glass solution is best suited to the soap. Good, unadulterated water-glass containing 37 to 38 per cent. of sodium silicate and showing at the same time  $38^{\circ}$  to  $40^{\circ}$  B. at  $77^{\circ}$  F. can be diluted with tank-lye of  $1^{\circ}$  to  $2^{\circ}$  B. to  $30^{\circ}$  to  $31^{\circ}$  B. This as a general rule will be most suitable, though sometimes a stronger solution can be used by dissolving three to four per cent. of crystallized soda in the water-glass itself. To the soaps, which after filling with water-glass become thick and glassy, add tale filling prepared with salt water (100 pounds of water, 100 pounds of tale, and 20 pounds of salt water of  $22^{\circ}$  B.). To those who have never filled in this manner it may seem very circumstantial, but after acquiring some experience, it will be at once seen whether the soap fills and how it fills. By adding to 50 pounds of soap 20 pounds of water-glass solution it will be seen whether everything is in accord, and give a chance to correct defects, as to every 50 pounds of soap in the kettle 30 pounds of water-glass solution are added, which corresponds to a yield of about 230 per cent., the remaining 10 pounds still wanting serving for correction. Care must be had that the soap does not undergo alteration or become frothy, which happens when the soap is too hot or is clumsily and too long crutched. To give the soap a chance to develop it, as well as the water-glass solution, must, however, show a temperature of from  $175^{\circ}$  to  $176^{\circ}$  F.

The finished soap should show all the characteristics of a boiled soap, and must not "die off" glassy, which is an indication that salt is wanting, or must not be wet, which is a sign of it being too sharp.

More experience is required for the following formula: Linseed or cotton-seed oil 300 pounds, tallow 150, cocoanut-oil 1000.

All these fats are also boiled to grain-soap, the 300 pounds of cotton-seed oil and 150 to 200 pounds of the cocoanut-oil being saponified upon caustic soda lye of  $15^{\circ}$  B. A strong paste-soap having been formed, 100 pounds of cocoanut-oil and 100 pounds of caustic soda lye of  $25^{\circ}$  B. are alternately added. The whole

is then allowed to boil thoroughly through and the soap is then salted out with 10 per cent. of common salt. The sub-lye is also drawn off or pumped out, and the whole again boiled upon scant lye and ground out by assisting with the crutch. All other directions previously given are also followed, only the soap must be kept hotter and in filling, fire has to be kept under the kettle or the soap heated by steam.

*Coloring of half-grain soaps.*—The colors most used are for *gray*, Frankfort black or pulverized animal charcoal; for *red*, bole, colcothar, and also cinnabar; for *blue*, ultramarine. Stir the coloring substance in warm water, add some salt, and color the soap boiling up finished until the tone of color comes out plainly. For filled soaps it is best to rub the coloring substance with melted palm-kernel oil and add it to the soap boiling up finished.

*Boiling of half-grain soap in general.*—At present, when lyes from caustic soda are not much more expensive than tank-lyes, the process of boiling half-grain soap is surer than formerly. If the content of caustic soda in the soda is known, it can readily be previously calculated how much salt and carbonated lye are required to obtain a favorable result. Should, however, irregularities occur in boiling, which can only be due to the salt, they can at once be corrected by the addition of strong caustic soda lye, which by penetrating into the combination destroys the effect of the salt. By then carefully contracting the soap with coconut or palm-kernel oil, a healthy base-soap is again immediately formed. Such corrections can, however, only be made with briskly boiling soap. The boiling of these soaps requires many years' practice and a thorough knowledge of the raw materials to be used. Like the fats the alkalies vary very much, and caustic soda must never be used twice in the same manner without considering its content.

In buying from large factories the per cents. given may, as a rule, be considered correct. To lye from high-grade caustic soda to be used for half-grain soap, a few per cents. of alkaline carbonate and also some salt may at once be added, such additions facilitating boiling. But to lyes prepared from caustic soda of a poorer quality, especially that of English origin, such additions



cannot be made. Caustic soda of 76 per cent. English = 126 per cent. German is sufficiently pure for our purposes. It is generally used for the fabrication of toilet soaps, but is actually not much more expensive than the poorer qualities, and where salt is cheap but the caustic soda has to be brought a long distance, it is recommended to buy the best, as the freight saved compensates for the difference in price.

The foreign salts in low-grade qualities of caustic soda are, to be sure, computed very low and at the same time are serviceable in the boiling of household soaps. With lye from such caustic soda, half-grain and grain-soaps can be at once boiled and also the fats combined to paste-soaps, the salts preventing a rough and quick combination, while lyes from high-grade caustic soda, especially from ammonia-soda; to be used for household soaps, always require an addition of at least 5 per cent. of calcined soda and 2 to 3 per cent. of salt.

Which process of boiling half-grain soap, whether the direct or indirect method or the preparatory boiling to grain and subsequent augmentation, is to be preferred depends on the condition of the fats and partially, also, on the arrangement of the factory. With pure fats, free from dirt and acid, the direct process is generally preferred.

From a combination consisting of 700 pounds of cocoanut or palm-kernel oil, 100 pounds of tallow or palm-oil, 200 pounds of cotton-seed or linseed-oil, and 1000 pounds of caustic soda lye of  $26^{\circ}$  to  $27^{\circ}$  B., a good half-grain soap, with a yield of about 200 to 205 per cent., can be prepared; but one with a yield of 225 per cent. can be obtained by the preparatory boiling to grain of all the fats and subsequent augmentation with 50 to 60 per cent. of water-glass.

Moreover, with the above combination a yield of 200 to 205 per cent. can be obtained by direct boiling without the use of heavy filling, by allowing the soap-cuttings to dissolve upon caustic lye of  $25^{\circ}$  to  $26^{\circ}$  B., to which 5 per cent. of salt has been previously added, and then introduce the palm-kernel or cocoanut-oil. About 50 pounds of cocoanut-oil are retained for future use, and the soap is shortened with salt or carbonated lye in case it is not bright, but rough. By now adding the reserved 50 pounds

of cocoanut or palm-kernel oil, the soap can be well regulated, and the result will be a product which boils light and high in the kettle and forms well in the frames.

If in boiling this soap by the direct method water-glass is to be used, not more than 20 to 25 per cent. of the fat (in this case 250 pounds) can be taken. The boiling of such soap is not difficult, but it is necessary to know the effect of the caustic soda lyes worked as well as the properties of the water-glass. Two methods are in use: Take for 100 pounds of fat either a determined quantity of caustic soda, varying, according to the content of the latter, between 19 and 23 pounds, or lyes varying between  $26^{\circ}$  and  $30^{\circ}$  B., and the alkaline content of which is accurately known. With such knowledge the latter method is just as sure and the lyes used are purer. Dissolve the soap-cuttings upon 850 pounds of caustic soda lye of  $27^{\circ}$  B., prepared from a high-grade quality of soda, then add the water-glass and allow the whole to boil through. If, however, for instance, the best quality of Greenbank caustic soda has been used for the preparation of the lye, 5 per cent. of crystallized soda and 5 per cent. of salt are at once added to the lye and allowed to boil through with it. No such addition is generally made to lyes from poorer qualities of caustic soda, and moreover they are mostly used  $1^{\circ}$  to  $1\frac{1}{2}^{\circ}$  stronger. If in dissolving the soap-cuttings much water has evaporated, it is replaced by adding about 50 pounds of it. The cotton-seed oil and tallow are then allowed to boil through, and the melted palm-kernel or cocoanut-oil having been introduced, the whole is crutched. 50 to 80 pounds of the palm-kernel or cocoanut-oil are retained for future use. A kettle sufficiently large to allow of the expansion of the soap to double its bulk is of great advantage. Immediately after the establishment of a complete combination between fat and lye, spontaneous heating takes place, and the soap in rising lifts up with it everything in the kettle. Endeavor to keep the soap in check by throwing, and as soon as it passes into quiet ebullition take samples. Be especially careful to see whether the soap has sufficient salt. If the samples are viscid and glassy, add salt solution until the desired effect is produced, and then only take the touch, *i. e.*, the proportion of lye, into consideration. A mild soap containing

salt allows of the water-glass being boiled with it without trouble, while a viscid soap, showing an excess of sharpness, frequently causes much vexation. All soaps containing water-glass must be quickly regulated before rigid combination is formed, as this can seldom be successfully broken with salt water, while the use of lye only increases the evil.

With the use of lyes of such strength as it has been necessary to recommend (800 pounds of lye and 250 pounds of sodium silicate for 1000 pounds of fat) it will be readily seen that nothing evaporates even if the yield amounts only to 210 per cent. With some attention it can be always so arranged that as much moisture is in the kettle as is required for the formation of the soap. Too much water with a large percentage of water-glass is an error, but too little is a still greater one. The entire correction for such soap can only consist of the addition of salt solution and a small quantity of crystallized soda, which are introduced before the entire quantity of fat is in the kettle, the final improvement being made by the addition of melted cocoanut or palm-kernel oil. The finished soap must boil light and high in the kettle, break into roses up to the rim, and the scorched particles torn from the bottom of the kettle by stirring, should be small and white.

Tank-lyes of various strengths can be more concentrated with caustic soda and then used for boiling half-grain soap with the above charge, but the process is always somewhat uncertain. It is advisable to concentrate the lye to 30° B. and to use only 800 pounds of it. Reserve about 100 pounds of cocoanut-oil for final correction and boil as previously given. Salt water can be added, but carbonates only in exceptional cases. If after the addition of salt water the soap boils too thin, introduce some pure caustic lye and fix the excess with melted cocoanut-oil added in small portions.

If the charge consists preponderatingly of fats readily forming combination, there are but few cases in which the soap-boiler is not forced to the preparatory boiling to grain. It is, however, not necessary if all the fats are previously melted upon water and only the clear portion is used for direct boiling, or if the fats are free from substances which might produce irregularities in the



soap. Preparatory boiling to grain is always best with bone-fat, glue-fat, and sedimentary oils, while expressed oils, palm-oil, and refined tallow can be directly boiled, the latter process being especially advisable in a factory having but one kettle. Where there are two kettles preparatory boiling does not involve extra labor, since before the soap-cuttings are dissolved the grain is also ready and its transfer to the other kettle is readily accomplished.

The use of a larger quantity of carbonates for soaps boiled by the indirect method is partially due to the fats and partially to the manner of boiling. The grain is a neutral soap formed from the fat with pure caustic lye, and the augmentation wanting consists mainly of carbonates. With a charge, for instance, of 400 pounds of bone-fat, 200 pounds of tallow, and 400 pounds of cocoanut-oil, the 600 pounds of fat = 900 pounds of grain, would require 300 pounds for augmentation. Now if 25 per cent. of water-glass is included in the charge, the lye required for it (5 per cent.) would still be wanting 120 pounds for augmentation, which could be supplemented by about half carbonates and half salt solution. It is, therefore, advisable at once to use for boiling, strong tank-lyes concentrated with caustic soda and to add three per cent. of salt. Such tank-lyes contain as much carbonates as required and the boiling itself proceeds regularly.

If with the same charge of 400 pounds of bone-fat or fats of the same consistency, 200 pounds of tallowy fats or palm-oil, and 400 pounds of cocoanut or palm-kernel oil, 20 to 25 per cent. of water-glass is to be added during boiling, pure caustic soda lyes will have to be used whether the process is executed in the direct or indirect way. With caustic soda of 60 to 62 per cent. carbonates should never be used, but 2 to 3 per cent. of salt may be added to the lye, but with caustic soda of 72 to 76 per cent. 5 per cent. of crystallized soda might be added in boiling to a finish and 5 to 6 per cent. of salt to the lye.

If the grain has been boiled upon caustic lye, it is dissolved together with the soap-cuttings upon 450 pounds of caustic soda lye of 25° B. The water-glass is then added, and after boiling the mass through, 30 to 50 pounds of salt are scattered over it. The melted cocoanut or palm-kernel oil is then introduced, re-

serving, however, 50 pounds of it for subsequent correction, and the formation of a uniform combination assisted with the crutch.

From all that has been introduced into the kettle, viz:—

Grain	.	.	.	.	.	.	.	900 pounds.
Cocoanut or palm-kernel oil	.	.	.	.	.	.	.	400 “
Water-glass	.	.	.	.	.	.	.	250 “
Lye	.	.	.	.	.	.	.	450 “
Total	.	.	.	.	.	.	.	<hr/> 2000 “

there will boil, under the most favorable circumstances, a soap with a yield of about 200 per cent., but by taking evaporation into the calculation and what has been absorbed by the dry soap-cuttings, the yield will be about 190 per cent. Hence 100 pounds of water would still be wanting for a normal half-grain soap, which quantity, and perhaps more, will have to be added before further evaporation can be thought of. Generally weak solution of salt or soda is also wanting, and by introducing it in small quantities the soap absorbs it and is improved, while by strong evaporation, after combination is once formed, the soap is disintegrated, and when salt and water-glass are once separated, contraction becomes difficult. Caustic lye for fitting may also still be wanting. It is, however, used only after the addition of the salt, lye of 20° B. being suitable for the purpose, as it attacks the combination less than stronger lye.

The finished soap should boil up high and light and break into roses over the entire kettle. It must, however, be sufficiently thick and the samples remain heaped up in a pile, otherwise it is too strongly fitted. After the addition of salt or solution of carbonate, the soap must always boil thin.

By taking 400 pounds of peanut-oil or cotton-seed oil, 200 pounds of tallow, and 400 pounds of palm-kernel or cocoanut-oil, and boiling by the direct method with 25 per cent. of water-glass, pure caustic soda lyes can also only be used. By bringing 900 pounds of lye of 27° B. into the kettle and dissolving the soap-cuttings upon it, then adding the water-glass and finally the peanut-oil or cotton-seed oil, the tallow and about 350 pounds of cocoanut or palm-kernel oil, there must be, when the soap boils up high, a thick, close mass in the kettle, which is so far regu-

lated with salt water that the samples do not "die off" glassy. If now the remainder of the cocoanut or palm-kernel oil be added, care must be had to evaporate the soap so far that it will be finished after the last fitting with oil. A soap with such a high percentage of water-glass may boil high and light, but evaporation takes place only to a small extent.

It may further be remarked that such soap creates trouble only when allowed to boil for some time without salt after combination is established, as the water-glass solution enters with the soap into that rigid, viscid union which can seldom be broken without serious consequences. Hence salt is always added either to the lye or immediately after combination is effected. If not certain whether the lyes contain salt, choose the latter way and add salt solution after combination is effected, there being no hurry with the carbonated lyes, which can always be subsequently incorporated.

If for the above charge the highest possible filling with water-glass (50 to 60 per cent.) is to be used, all the fats are first boiled to as neutral a grain-soap as possible by bringing into the kettle first the cotton-seed oil, bone-fat, tallow, and about 100 pounds of the palm-kernel oil, together with about 800 pounds of caustic soda lye of  $15^{\circ}$  B. A good combination being established, add 400 pounds more of caustic lye of  $20^{\circ}$  B., and later on the remaining cocoanut-oil. Then add caustic lye of  $28^{\circ}$  B. until a clear paste-soap showing a strong touch boils in the kettle. Boiling is now interrupted and the soap separated with 10 per cent. salt solution.

Now sprinkle 100 pounds of salt dissolved in water over the soap and continue boiling, when, as a rule, a clear soap will show itself, which, if necessary, is somewhat re-ground, so that a bright grain is formed, which is tightly covered and allowed to stand over night to settle.

The sub-lye can seldom be sufficiently removed by drawing off or pumping out, and it is therefore best to ladle the grain, after testing whether it will fill, into a special kettle provided with a stirring apparatus. The water-glass which serves for filling is diluted with tank-lye of  $1^{\circ}$  to  $2^{\circ}$ . How far this can be done has to be ascertained by a test; generally it is, however, reduced to



28° or 30° B. Salt solution, hot water, and solution of crystallized soda must also be kept in readiness, so as to assist with them if necessary. The soap must become quite thick and curly, not "wet," but be quite dry. The filling may be heated to 167° F., and if the soap becomes cool a little fire may be started to warm it, but boiling is not permitted. The finished soap must allow of being readily stirred and resemble a dry grain-soap. Viscid soap is improved by salt water and soda solution.

A variety of half-grain soap, in which the yield is of little consequence, is prepared with the use of fuller's fat, wool fat, sedimentary oils, and other cheap fats.

Fuller's fat, though nearly pure fatty acid, gives but a moderate yield, there being few varieties of the crude fat which give over 135 per cent. of soap with a firm cut, which is partially due to the fat containing much dirt.

Direct boiling is not available with crude fuller's fat, it being, of course, different with the white fat purified by distillation—the so-called solid, white olein.

Boiling to grain is the more time-consuming the more colored the fuller's fat is, and if the process is to be entirely sure it cannot be done without ladling into another kettle, there being no pump or discharge-pipe which will remove the lye and dirt settling on the bottom of the kettle. The boiling may, however, be shortened by boiling out all the sub-lyes with fuller's fat and preserving the resulting grain for a large boiling, when the pumping out or drawing off of the sub-lye in boiling to a finish will suffice.

The consistency of the fat must also be taken into consideration. The manufacture of a soap of a firm cut would, for instance, be scarcely possible from liquid fuller's fat. Such fat originates from soap in the preparation of which linseed-oil has been used, and is generally worked, in connection with other fats, into soft soap. Crude fuller's fat of the consistency of bone-fat is derived from natural grain-soap or from hard bar-soap. Such fat may be improved by heating and ladling it into a barrel, when the gray-white or yellowish, solid, fatty acid separates, from which the oleic acid can be readily filtered off.

Fuller's fat is best saponified with tank-lyes not entirely

caustic, commencing with a lye of  $12^{\circ}$  to  $13^{\circ}$  B. and finishing with one of  $20^{\circ}$  to  $21^{\circ}$  B. It may be allowed to boil in paste, though salt can also be at once added to the lyes. In the first boiling strong touch must be kept up, whereby the fat is decolorized and the sub-lye acquires a dark-brown color. This has to be repeated several times if the fat is to be entirely decolorized by boiling, and sometimes it will not even succeed then. The fat is generally boiled upon three waters, the sub-lye being then lighter, and this, as a rule, suffices. Upon the third water the soap has to be boiled so far that, if necessary, it will form a solid grain-soap.

For the fabrication of half-grain soap the clear grain is lifted off, brought into the kettle with caustic lye of  $20^{\circ}$  B., and allowed to boil through. Coconut or palm-kernel oil is then added until all the lye is fixed and a uniform soap shows itself in the kettle. If the kettle now be covered, the thick soap will be observed to liquefy in about one hour. It is then evaporated until a sample shows the degree of hardness the finished soap is to have.

Wool-fat is another cheap fat. It is obtained from crude wool and has a red-yellow color. Boiled by itself it does not give soap, but can be utilized for soaps with a firm cut in connection with fuller's-fat, sedimentary oils, and poorer qualities of bone-fat. Sedimentary oils from rape-seed oil, linseed-oil, etc., can also be boiled to grain only with better qualities of fat. The object being to work these cheap fats into soap of good appearance, they in connection with other fats on hand are first boiled to grain. In consequence of the strong evaporation required this grain-soap presents a mean appearance and yields a lather with difficulty. By contracting it, however, with coconut-oil, an actually beautifully mottled soap uniform all through is obtained, and, besides, by this mode of boiling the soap-boiler has it in his power to impart to it any desired degree of hardness. The soap must, however, be sufficiently evaporated, and salt and, sometimes, solution of alkaline carbonate added. It will be readily understood that the yield is a very moderate one, but the soap is as good for household purposes as curd-soap, and will be readily bought after the consumer is once accustomed to its appearance.

## CHAPTER XV.

## MANUFACTURE OF SOAP BY THE COLD PROCESS.

*Fats for saponification by the cold process.*—Under saponification by the cold process is understood the method in general use at the present time of preparing soaps, and especially the cheaper toilet soaps, by simply stirring together melted cocoanut-oil, as well as palm-kernel oil, with exactly the quantity of lye required for saturation. The process is based upon the property of the various kinds of cocoanut-oil to form at a low temperature a combination with strong lye, which, by subsequent spontaneous heating in the frame, yields a solid white soap distinguished by giving an abundant lather. Frequently a certain per cent. of tallow, lard, olive-oil, or castor-oil is used, and though cocoanut-oil saponified by itself gives the purest and finest colors, both in the white and colored product, the addition of one of these fats is advisable in all cases where a milder and more durable product than from cocoanut-oil alone is desired. Such soaps, particularly when prepared with a considerable percentage of tallow, resemble very much in appearance milled toilet soaps, and when well and carefully made can frequently be alone distinguished from them by the fracture, which is amorphous in soaps prepared by the cold process, but crystalline in milled soaps.

The manner of preparing soap by the cold process is the same whether tallow and other fats and oils are used in connection with cocoanut-oil or not. There is but a slight deviation as regards the temperature of the fats to be saponified and the quantity of lye required, it being only necessary to observe that of the fats here in question cocoanut-oil requires the largest amount of lye for complete saponification. Next are tallow, lard, and olive-oil, which require about the same amount of lye; castor-oil needs least. It is, of course, understood that only fresh and pure olive-



oil can be used, and that all the other fats should be as fresh and pure as possible, and especially not rancid, as otherwise the soaps would be very much inclined to rancidity. As regards the temperature it may be set down as a rule that the higher the melting point of a fat to be saponified by the cold process the higher the temperature must be kept during the operation. If, for instance, tallow could be worked by itself, it would have to be kept at from  $140^{\circ}$  to  $144.5^{\circ}$  F., while in connection with one-half to two-thirds of cocoanut-oil a temperature of from  $104^{\circ}$  to  $108.5^{\circ}$  F. suffices for the first case and one from  $90.5^{\circ}$  to  $95^{\circ}$  F. for the latter.

For cocoanut-oil alone, as well as when worked in connection with lard, castor-oil, and olive-oil, the temperature in summer should not exceed  $79.5^{\circ}$  F., one of  $64.5^{\circ}$  F. being frequently sufficient, and in winter  $90.5^{\circ}$  F. If tallow is used, a somewhat higher temperature is required, especially in winter, though it never need exceed  $113^{\circ}$  F.

It remains to say something in regard to cocoanut-oil. The three principal varieties at present known in commerce are Ceylon, Sydney, and Cochin China oils, the latter yielding the whitest and most delicate soaps, and will have to be used where whiteness, durability, and freedom from odor are especially demanded. The other oils, being cheaper, are employed for cheaper and colored and perfumed soaps, though for the finer qualities of colored soaps, Cochin China oil is preferable, as the colors will turn out more delicate and beautiful. Cochin China oil being generally brought into commerce in a fresher state than the other kinds, saponifies slower and more regularly, almost all disturbances in the cold process of saponification being due to free fatty acids, which frequently cause a premature thickening of the soap, so that before the addition of the last lye the mass can no longer be worked with the crutch. Old Ceylon and Sydney oils are especially rich in such free fatty acids. To remove this evil it is best previously to refine such oils with strong lye, the process for which will be described further on; though frequently even this radical remedy is of no avail, especially with very old oils, and it is then advisable not to use them for cold saponification.

All other disturbances and interruptions in the cold process of

saponification, such as the soap becoming thick and gritty, the formation of grain, and partially, also, the gray color of the soap, are also due to a greater or smaller content of free fatty acids in the oil. Frequently these evils can be partially prevented by keeping the oil during saponification at as low a temperature as possible, even as low as  $71^{\circ}$  to  $77^{\circ}$  F., according to season of the year. There is no cause for alarm even if, in the beginning, after the addition of the lye, the mass partially congeals so that lumps are formed, because when the soap becomes more strongly heated by the lye, these lumps re-dissolve and the soap again becomes smooth and gradually thinner, so that stirring can be continued to the normal thickening. Soaps thus treated are very mild and white; this result, however, being reached only by vigorous and continuous crutching. If, however, the soap becomes thick and grainy even with this treatment, or a pure white color and great durability of the soaps are especially demanded, the process of refining with strong lye, above referred to, must be resorted to. And even if the soap-boiler is not forced to refine the oil, previous washing with salt water to remove particles of dirt and mucus mechanically fixed in the oil is absolutely necessary. This is effected in the following manner:—

Bring into a shallow kettle filled about two-thirds full with the cocoanut-oil to be purified, a sufficient quantity of salt water of  $15^{\circ}$  to  $18^{\circ}$  B., so that the oil, when melted and floating upon the salt water, can be conveniently ladled out. After melting the cocoanut-oil, bring it to the boiling point and keep it there for at least half an hour, removing the scum constantly forming on the surface. The operation is finished when only pure white froth comes to the surface. Now shut off the heat, cover the kettle, and allow the oil to stand a few hours or, still better, over night. The next morning draw off the clear oil from the sediment and dirt.

Where steam is available it is not necessary to introduce water into the kettle, since it is formed by the steam condensing. The required salt (about 3 to 4 pounds to 100 pounds of oil) is brought into the kettle together with the oil.

Refining or bleaching the oil with lye is carried on in a similar manner. First melt the oil, then bring the required quantity of

water into the kettle, and after heating the oil to the boiling point, add carefully, so as to prevent boiling over, 3 to 5 pounds of lye of  $38^{\circ}$  to  $40^{\circ}$  B. for every 100 pounds of oil. After thoroughly boiling the mass until it may be expected that the lye is completely saponified, salting out with dry common salt is commenced and continued until a dirty gray and frothy grain rises from below to the surface, which is removed as soon as it appears. This grain must be so compact as to remain upon the skimmer; if it is so fluid as to pass through the holes in the skimmer more salt is required. The operation requires quick work, because as soon as the froth is allowed to cover the entire surface so that the oil cannot break through, boiling over cannot be prevented, and the addition of water or lye would only still more promote combination, thus increasing the evil. For this reason a very moderate fire should be kept up during this stage and the froth not allowed to collect. The appearance of a pure white froth indicates the end of the operation, when the kettle is well covered and the oil subsequently treated in the same manner as given for purifying with salt. After settling, the oil is, however, not clear, but milky white and turbid. In cooling a thin pasty film frequently forms upon the oil. It is of no use to remove this, since it does not injure the quality of the oil and only forms again. Oil treated in this manner saponifies slowly and with difficulty, so that, for instance, 200 pounds of oil frequently require half a day and sometimes still longer for thickening. This is, however, a good sign, as oils difficult to saponify yield the whitest and most durable soaps. This process of refining or bleaching of cocoanut-oil is, besides, the only means known at the present time of preparing soaps as inodorous as possible.

From what has been said it will be seen that, as far as possible, only the freshest cocoanut-oil should be used for saponification by the cold process, in order to avoid the loss incurred by refining and to be able to produce beautiful, durable, and white soaps without taking up much time for preparatory work. Where opportunity is offered to select the cocoanut-oil, it is recommended to choose such as, with a hard and a quite transparent white fracture, shows the least odor of rancid butter. Smeary oils with a rancid butter odor and of a greenish or gray color may be at



once designated as old oils containing much free fatty acid, which will not yield beautiful and durable soaps without previous refining. The odor of fresh cocoanut-oil, especially of Cochin China-oil, is peculiarly aromatic and nutty.

As previously mentioned, for the better qualities of soap it is advisable to use tallow, lard, castor-oil, or olive-oil in connection with cocoanut-oil, the results obtained with the first two fats being especially desirable, they making the soaps milder without injuring their hardness and feel, and 50 per cent. of them may be added to the cocoanut-oil without disturbing the process of saponification, it being only necessary to reduce the proportion of lye, as these two fats do not require as much as cocoanut-oil alone.

*Lyes for the cold process of saponification.*—The lyes formerly used for the fabrication of soap by the cold process were generally prepared from calcined or crystallized soda subsequently causticized with lime. Lyes of  $12^{\circ}$  to  $15^{\circ}$  B. were obtained in this manner and then evaporated to  $40^{\circ}$  B. This gave a very good and serviceable material, but the evaporation was connected with great loss of time, labor, and fuel. In more modern times the lyes required for the manufacture of toilet soaps are generally prepared from caustic soda,\* and this is to be the more recommended as the process is very quick and sure.

The use of distilled water or rain-water in the preparation of the lye is of great advantage. Where, however, no other water but well or river water is available, it should be boiled and allowed to settle before use.

After setting the lye to the right degree B., cover the reservoir with the lid and bags, and allow it to stand quietly a few days for the particles of sediment and dirt to subside. It is further to be remarked that in setting the lye it must not be hot, or even warm, as lye when cooled off shows always more degrees than when hot. In measuring by Baumé's areometer  $63.5^{\circ}$  F. may be taken as the normal temperature.

After remaining for a few days in the closed reservoir the lye has acquired a clear appearance. To perfect it still further and

\* For the preparation of lye from caustic soda see p. 220.

to remove the smallest particles of dirt or sediment, it is filtered through a glass funnel, filled with glass-wool, into large clean glass-balloons.

For soaps by the cold process it is best to use only high-grade lyes, since non-success is frequently due to the large content of carbonates and other salts in the weak lyes, which results in spongy, soft, and badly-combined soaps, bad colors and inclination to sweat in winter and on rough days. Good soda, on the other hand, produces clear, transparent, and thoroughly combined soaps of a white and delicate appearance, and firm and solid to the touch. A moderately filled soap prepared with good soda may frequently excel in appearance and quality a non-filled soap prepared with poor soda. Care should, therefore, be had in the choice of the soda and the preparation of the lye, and the soap-maker should not be afraid of a little extra expense for an article of good repute, for in the manufacture of household as well as toilet soaps, soda is the basis of all. It is far easier to prepare a product of a good, or at least medium, quality from poor oils or fats and good soda than *vice versa*. All defects and blemishes of the soap, if there be no direct evidence to the contrary, can in nine cases out of ten be traced to the soda or the proportion of lye used. As a precaution it is therefore best to use 75 to 76 per cent. caustic soda for all soaps by the cold process, and allow one pound of lye of 38° B. prepared from it for every two pounds of cocoanut-oil.

On the other hand, for soaps by the warm process, to which belong, for instance, all boiled cocoanut-oil soaps, as well as transparent or glycerin soaps, it is advantageous to use 70 to 72 per cent. caustic soda; since these soaps require a certain content of salts found in the low-grade varieties of caustic soda. As, however, this content of salts exerts no effect in the saponification, though it counts in weighing the degrees of the lye, it is necessary to use the lye prepared from such soda at 40° B.

*Filling for soaps by the cold process.*—By the word “filling” is understood the art of being able to produce the soap cheaper by mechanically mixing with it less expensive substances than the soap itself. This is not the place to enter into a justification or condemnation of this method, though we may be allowed to say

that no doubt every soap-maker would prefer to manufacture pure and unfilled soaps provided he received a corresponding price. However, this not being the case, cheap soaps being generally demanded and bought in preference to expensive ones, the soap-maker is forced to use means of cheapening his product in order to compete with others.

The object of filling is partially to increase the quantity and partially the weight of the soap. The agents used for increasing the quantity are salt water, potash solution, and sugar solution; and those for increasing the weight, talc and water-glass.

Salt water is applied as a solution of  $15^{\circ}$  B. of pure common salt in pure water. After removing the scum and dirt floating on the surface by means of a skimmer, the solution is added to the soap when it seems to be thoroughly combined and before bringing it into the frames. Much salt solution cannot be used for cold-stirred soaps, as it disturbs the combination and frequently causes entire failure. It besides possesses the property of producing soaps which are moist on the cut surface, and in drying in the store-room lose their shape and become hard as stone. For this reason salt solution as a filling material for cold-stirred soaps should be rejected, and especially as such soaps are generally inclined to sweating and consequent soaking of the wrapper.

The use of *potash solution* as a filling material requires good caustic lye for the saponification of the fats and oils, because it decreases causticity, the result being spongy and soft soaps. With good caustic lye it is, however, an excellent material for making soaps filled with water-glass more transparent and preventing them from becoming hard. It is best used at from  $15^{\circ}$  to  $18^{\circ}$  B., and generally in connection with water-glass.

Of *water-glass* two principal varieties are found in commerce—soda and potash water-glass.\* For the manufacture of toilet soaps, soda water-glass, or sodium silicate, is chiefly used, and besides it is cheaper than potash water-glass, or potassium silicate.

Water-glass represents, in a certain manner, a finished soap, with slight detergent power, and readily combines with the soap

\* See page 218.



to be filled. It is used from 30 to 40° B. strong, being reduced to 30° B. by the addition of water. Though soaps filled with water-glass retain some of their transparency, they become almost petrified by storing, by the moisture drying out, while the silicic acid remains. This evil is overcome, as above mentioned, by using potash solution in connection with water-glass. Water-glass possesses also the property of weakening the fit of the soaps, so that it frequently happens that soaps strongly filled with water-glass yield oil during the heating in the frame which spoils the soap. Lye is therefore mixed with water-glass in the proportion of 1 pound of lye of 38° to 40° B. to 5 pounds of water-glass. When the soap is thoroughly combined, the water-glass is added, and, after several times vigorously crutching the mass, quickly brought into the frames.

Solution of sugar is used as a filling material to impart to the soap transparency and clearness, though it is apt to make it soft and sticky, and renders it soon odorless and then rancid. Solutions of various strengths are used—for instance, 1 pound of sugar to 2 of water, 1 pound to 3 of water, and so on.

All the filling materials mentioned, with the exception of sugar solution, make the soap more or less sharp, and soaps thus prepared cannot be recommended to persons with sensitive skins. Talc, however, is free from this evil, and rather makes the soap milder and innoxious to the skin, but has the disagreeable property of injuring the appearance of the soap, the color of the latter being never so beautiful, clear, and pure as that of soap filled with the other materials, though, at the same time, it does not become so hard. White soaps filled with talc turn gray, while colored ones lack the freshness and the brilliancy so much esteemed.

Talc is applied by finely pulverizing it, sifting it through a fine sieve into the liquid fat or oil, and uniformly incorporating it by vigorous stirring. The lye is then added, and the process carried on as with other soaps.

Other additions to the soap, such as glycerine, etc., cannot be considered as filling, since they are intended for a specific purpose, as, for instance, in medicated soaps, to produce a certain medicinal effect upon the skin, etc.

*Marbling of soaps prepared by the cold process.*—The marbling of these soaps presents no difficulties, requiring only some experience. It is done in the following manner:—

Before stirring the soap together, weigh off the required quantity of coloring material into the rubbing dish, and triturate it with some olive oil. When the soap is thick enough for pouring out (for marbling it should be somewhat thicker than usual), bring a few pounds into the rubbing dish, and mix it thoroughly with the color. Now, while one workman uninterruptedly stirs the small quantity of soap thus colored to prevent its too rapid cooling, another ladles a sufficient quantity of soap into the frame to cover the bottom. Upon this is poured in a thin stream a portion of the colored soap in lengthwise and crosswise streaks, and then more soap is introduced into the frame, so as to cover the color. Colored soap is again poured upon this in the same manner as before, upon which comes again a layer of soap from the kettle, and this is alternately continued until the frame is full. For distributing the colored soap a small crutch provided with holes is used, which is several times drawn up and down in the soap, but every time at a new place. Finally, large or small circular or annular figures are drawn through the soap from one end of the frame to the other by means of a rod pushed down to the bottom. With some experience, an irregular but nevertheless beautiful marbling is thus produced.

A more regular marbling is obtained in the following manner:—

When the soap is ready to be poured into the frame, color the surface of the soap in the kettle, so that the coloring substance mixes with the soap, without, however, penetrating too far down. Then, by a quick turn, pour the contents of the kettle into the frame, whereby the colored soap will be quite uniformly distributed, the distribution being completed with the crutch. Streaks are then drawn lengthwise, about the width of a finger apart, from one end of the frame to the other by means of a rod, commencing, however, always on one end of the frame without returning in the streak just made.

The manner of coloring these and other toilet soaps will be given in Chapter XVIII., treating of toilet soaps.

## CHAPTER XVI.

## SOFT SOAPS.

By soft soap is understood a soap prepared chiefly from potash lye and oil, of a soft, salve-like consistency, and containing much more water than hard soap. It is not a simple alkaline sebate, but a solution of an alkaline sebate in a mixture of carbonated and caustic lye. As neutral potassium oleate yields a viscid, gummy, and turbid product, it is necessary to add to it, in order to obtain the transparent soap of commerce, a solution of potassium hydroxide and carbonate, or to substitute for the latter one of allied salts. By depositing itself between the atoms of soap, the potassium carbonate, added in a corresponding proportion, breaks up the viscosity of the soap and forms it into a plastic, transparent mass, while it also possesses the property of combining water with the soap.

It is frequently stated that the glycerin contained in the oil and fat takes an active part in the process of saponification. Such, however, is not the case, it contributing at the utmost only to the transparency of the soap.

The principal fatty substances—train-oil and hemp-oil—which were formerly used in the manufacture of soft soap are but little employed at the present time, linseed-oil, German sesame or cameline-oil, rape-oil, cotton-seed oil, peanut-oil, sesame-oil, as well as oleic acid, tallow, palm-oil, lard, and horse-fat being substituted for them. On account of their low price and other good qualities, linseed-oil and cameline oil are, however, the principal materials used.

The success of the soap depending chiefly on the lyes, it is necessary to prepare them with the greatest care, the principal condition being to determine whether they contain correct proportions of caustic alkali and alkaline carbonate, or, as it is technically called, are “correct in lime.”



For the saponification of 100 pounds of oil or fat about 36 pounds of 96 per cent. potash causticized by lime are generally required. If, however, the soft soap is to be filled, a considerable quantity of lye is necessary for saturation, so that 40 pounds of 96 per cent. potash must be allowed for 100 pounds of oil.

For boiling, lye of an average strength of  $23^{\circ}$  to  $24^{\circ}$  B. is generally used, 155 to 160 pounds of lye of  $24^{\circ}$  B. sufficing for 100 pounds of oil. Lyes taken directly from the tanks are not even in lime, the latter varying very much in lyes of  $30^{\circ}$ ,  $24^{\circ}$ , and  $18^{\circ}$  B.; the higher the degree of the lye the lower in lime it is. It being, however, absolutely necessary in boiling to keep within determined proportions of caustic alkali and alkaline carbonate, correspondingly weaker and stronger lyes are used; for instance, about 2 parts of lye of  $20^{\circ}$  B. and 1 part of lye of  $30^{\circ}$  B., by which a suitable average lye of about  $24^{\circ}$  B. is obtained.

The manner of preparing the various kinds of soft soap occurring in commerce being the same and carried on according to the same rules, they can only be divided into the following four varieties, according to the fatty substances used and whether potash lye alone is employed or soda lye in connection with potash lye. 1. Smooth transparent soap. 2. Transparent soap with natural grain (natural grain-soap). 3. Transparent soap with artificial grain (artificial grain-soap). 4. Soap with a mother-of-pearl lustre (silver soap, elaidin soap). All these varieties demand a different treatment in the various seasons of the year, in order to present always the same appearance; it being of special importance to choose a different combination of fats for each season of the year, to pay special attention to the correct proportion of causticity of the lyes, the degree of moisture and the fit of the soaps, and to use the correct proportion of soda for smooth, artificial grain, and silver soaps.

1. *Smooth transparent soft soap*.—For these soaps, which occur in commerce as so-called crown soaps of various colors (black, brown, green, etc.), linseed-oil or cameline-oil is generally used in winter, and sometimes train-oil and hemp-oil, these oils being nearly free from stearin and little inclined to congealing. In the warm season of the year, besides the mentioned oils, a considerable quantity of cotton-seed oil, rape-oil, peanut-oil, sesame-oil,

and olive-oil is worked to impart to the soaps a better consistency and durability. On account of its low price, linseed-oil is much used at the present time for the manufacture of smooth soft soap. Though a pure potash soap prepared from it will stand any degree of cold, it will liquefy at a higher temperature, so that to overcome this evil a certain quantity of potash lye is replaced by soda lye. Greve and J. Gentile made various experiments in regard to the amount of soda allowable for soft soap, and showed that a good article could be obtained in summer by the use of 3 parts of potash lye and 2 parts of soda lye, while with the use of equal parts of potash and soda lye the soap becomes turbid and unsalable, the same occurring when the soda contains much common salt and the potash much potassium sulphide or chloride. At the present time one-fourth to one-third soda lye is generally used during the hot season of the year, which is, however, gradually reduced (to one-fifth, one-sixth, etc.) with the approach of colder weather, and entirely omitted when frost sets in, potash lye alone being then used. An allowance must, of course, be made for the soda naturally contained in the potash. It may be further remarked that soft soap, in the preparation of which at a low temperature too much soda lye is used, becomes short and friable, separates lye, and acquires a turbid, bad appearance, and in freezing weather this evil may already occur with the use of one-fifth soda lye. Furthermore, while the clearness of the soap suffers the more, especially under the influence of cold, the greater the content of soda, a smaller yield is also obtained and a somewhat more expensive product. By frequent weighing of different boilings it has been ascertained that a soap prepared with 6 parts of potash lye and 1 of soda lye yields at the utmost 230 pounds of soap from 100 pounds of linseed-oil, and that the yield diminishes in a certain proportion with a higher percentage of soda lye.

Suppose a soft soap boiled from linseed-oil and pure potash lye yields, when sufficiently evaporated, 235 pounds, and a pure soda soap only 190 pounds, the yield with the use of potash and soda lye can be readily calculated.

1 part soda lye . . . . .	= 190 parts
6 parts potash lye @ 235 . . . . .	= 1410 “
<hr/>	
7 “ in . . . . .	1600 “ = 228½
Or 1 part soda lye . . . . .	= 190 parts
4 parts potash lye @ 235 . . . . .	= 940 “
<hr/>	
5 “ in . . . . .	1130 “ = 226
Or 1 part soda lye . . . . .	= 190 parts
2 parts potash lye @ 235 . . . . .	= 470 “
<hr/>	
3 “ in . . . . .	660 “ = 220

Resin (5 to 15 pounds to 100 pounds of oil) is frequently added to smooth, transparent soft soaps, which not only renders the soap cheaper but also gives it a fine lustre. The resin is either at once added to the oil in the kettle and saponified with it, or is introduced with the required quantity of lye of 30° B. (about 92 pounds to 100 pounds of resin) into the soap boiling up finished and combined with it by crutching. The latter process is chiefly applied, as it gives lighter soaps and a somewhat larger yield, though the soap has again to be fitted after adding the resin. On the other hand, by boiling the resin together with the oil, the soap becomes somewhat darker and the yield somewhat less, but the resin is better incorporated with the soap, and a second fitting being not necessary the operation takes less time and is surer.

The use of resin rendering the soap considerably softer, it is necessary to use besides potash lye a corresponding quantity of soda lye of 24° B., and the more the warmer the season of the year.

*Smooth soft soap from linseed-oil.*—In many parts of Northern Germany a smooth soft soap of a red-yellow, brown, or green color is manufactured. In winter the soap is boiled with potash lye alone and quite strongly fitted with potash solution of 28° B., which not only protects it from freezing but somewhat increases the yield. If resin is to be worked in connection with the oil, it is generally saponified with soda lye. In the warmer season of the year oils and fats containing more stearin, such as cotton-seed oil, peanut-oil, oleic acid, horse-fat, etc., are frequently worked in connection with linseed-oil, whereby the soap acquires a better



consistency and greater power to resist heat. A portion of the potash lye is also replaced by soda lye (one-fifth to one-third). Care must be exercised in the addition of oils containing stearin; while in summer as much as 20 to 25 per cent. of cotton-seed oil or oleic acid may be used, the quantity must be considerably reduced with the approach of cooler weather, and the addition entirely stopped before cold weather.

To prepare the necessary lye for the saponification of 1000 pounds of linseed-oil it is best, in order to be provided for all emergencies, to use 500 pounds of 96 per cent. potash and 216 pounds of lime. The lye obtained by the first lixiviation is generally set at 28° B. and that from the second at 20° B. Lye of an average strength of 23° B. is generally used, of which for the saponification of 100 pounds of linseed-oil about 170 pounds (hence 100 pounds of 20° B. and 70 pounds of 28° B.) are required.

The boiling of the soap is carried on as follows: In the evening 1000 pounds of linseed-oil and about 400 pounds of lye of 20° B. are brought into the kettle. The next morning the fire is started and the oil and lye are from time to time crutched through. When the mass is sufficiently hot an emulsion will be formed by crutching, the contents of the kettle begin to rise somewhat and combination be established, the latter being recognized by a sample not yielding lye and running from the spatula like a thick homogeneous ointment. A thorough and intimate combination being established, 300 pounds more of lye of 20° B. are immediately added, and as soon as they have combined with the rest, 300 pounds more. The fire is now increased, and about 650 pounds of lye of 28° B. are introduced in three or four portions, care being had to allow each portion to combine before adding the next. When all the lye has been brought into the kettle, which will require about one hour, a dark but quite clear mass (paste) shows itself under a light cover of scum. Though a sample dropped upon glass is at first clear, it is still very thin and draws threads, and on cooling acquires a turbid appearance and becomes jelly-like. The scum upon the surface of the soap and the condition of the sample indicate that the soap contains too much water. To remove this the fire is increased and the

soap thoroughly boiled and evaporated. After boiling for some time the soap becomes clearer, thicker, and shorter, the scum upon the surface gradually disappears and the soap begins to "talk,"\* to boil in large laminæ and to break into roses.† At this stage the soap when thrown with the spatula will show no water bubbles, but runs off in elevated streaks and will break from the spatula without drawing threads. When the moderately boiling soap shows these signs and no more scum is seen upon the surface and a sample upon glass forms a small heap without many pearls of froth or bubbles of air, evaporation has been carried sufficiently far, and fitting of the soap, *i. e.*, testing it as to whether the oil has been completely saponified by the alkali, is proceeded with. This is ascertained with greater ease and more certainty with soft soaps than with soda soaps as the latter become turbid on cooling.

Every drop of a pure oil soap shows exactly the same characteristics and behavior as the entire contents of the kettle, from above to below, would show only after a few days, and therefore the most accurate observations can be made on a clear sample. If the 1650 pounds of lye have sufficed for the saponification of the 1000 pounds of oil, a sample dropped upon glass is clear and translucent, and, when held against a dark background, shows "flower"‡ and a small ring of lye.§ If the sample becomes turbid and whitish, and glides or is slippery on the glass, it is an indication of the soap containing too much lye. This is remedied by gradually adding oil to the boiling soap, until a sample shows the above characteristics of well-fitted soap. It is best to emulsionize the oil with some weak lye before adding it, as in that state it combines better with the soap. If, on the other hand, the sample upon the glass is clear, but without "flower," it is a sure sign that lye is wanting.

After thoroughly examining and observing the samples, they

\* The technical term for the noise produced by the steam developed on the bottom of the kettle in forcing its way through the thick soap.

† The thick soap on the place where the steam from below forces its way through resembles an expanding rose.

‡ The appearance of a striped skin or lye-veins on the surface of the sample.

§ A fine hair-white ring surrounding the sample.

are placed in the cellar, where the soap is, later on, to be stored, and in which an even temperature of from  $50^{\circ}$  to  $59^{\circ}$  F. should prevail. The samples of soap intended for the warm season of the year are generally placed near the ceiling of the cellar, and those for the colder season upon the floor. Soap for the warmer season must be slightly fitted to "small flower," and if the sample, after remaining half an hour in the cellar, is short, firm, and clear, it is correctly fitted. For winter soaps the fresh sample may show a somewhat stronger "flower," and the one cooled upon the stone-floor of the cellar a considerable ring of lye. If, however, the sample cooled in the cellar is clear around the edge, and shows a turbid, milky point, the so-called "fat-gray" in the centre, some lye is wanting. This is remedied by carefully adding to and boiling with the soap small portions of lye, until a sample dropped upon glass and cooled in the cellar is entirely clear.

When the soap boils up finished, 5 to 15 per cent. of resin, with the corresponding quantity of lye of  $30^{\circ}$  B. (92 pounds to 100 pounds of resin), may be added, with constant stirring, and keeping the fire-door open. The soap must then be again fitted. Soda-lye is generally used for the resin. We would remark that in summer it is advisable not to exceed 10 per cent. of resin, though in winter, and for soaps strongly filled with flour, somewhat more can be used.

*Particular remarks.*—Though the previously mentioned proportion of lime will, as a rule, be found suitable, instances may arise where such is not the case. The "proportion of lime" is the chief trouble of the inexperienced soap-boiler, and causes him fresh anxiety with every boiling. It may happen that the lye is actually somewhat too caustic, or contains too much carbonate, and that this becomes perceptible in the soap-mass, so that a correction is desirable. Frequently, however, the opinion that the proportion of lime is not correct is based upon a deception, and the unsatisfactory appearance of the sample upon the glass is due to other causes, especially to insufficient evaporation and want of lye, which misleads the inexperienced soap-boiler, and makes him think the lye is "too high in lime." He will, however, acquire



the necessary experience and accuracy by close observation and long practice.

That the soap is too high in lime is recognized by its boiling heavily farther down in the kettle, rising only with a very strong fire, and running from the spatula in broad, viscid streaks, and a sample dropped upon glass being turbid and gummy after cooling. Normal boiling of the soap is restored by successively adding potash-solution of  $26^{\circ}$  B., until the samples dropped upon glass are short, firm, clear, and pliant. Should, on the other hand, the soap be "too low in lime," it boils up high, and rises over, so that it can be scarcely curbed, has a thinly-fluid, aqueous appearance, and a sample dropped upon glass breaks up. This is best remedied by an addition of pure caustic potash-lye, or, when the soap does not contain too much soda, of caustic soda-lye of  $24^{\circ}$  B., and in summer also by oleic acid. It is, however, always advisable not to be too hasty in making corrections in the proportion of lime, but only after being convinced of the necessity by quite a strong evaporation of the soap.

Should the soap at the commencement begin to thicken, then it is wanting in lye, which must be added at once. But if the soap has once become thick, it is difficult to liquefy it, notwithstanding the addition of lye, and some lumps will not dissolve before an excess of lye has been added, with vigorous boiling. This causes, however, no injury, as the excess of lye can be readily overcome near the end of the operation by the addition of some oil.

Great attention must be paid to the sufficient evaporation of the soap, for a product containing too much water remains soft and on storing becomes turbid and thin and draws threads. The soap must be evaporated until no more scum is visible upon the surface, and samples dropped upon glass show but few pearls of froth and are sufficiently clear and firm.

Fitting of the soap is also of importance for its future behavior. Soap too slightly fitted becomes turbid, soft, and liquid in the barrels, while when too strongly fitted it becomes short, friable, and eventually separates lye on the surface. In fitting the soap the season of the year at which it is to be sold must be especially taken into consideration. In the cold season of the year it must be

strongly fitted to prevent it from freezing ; in summer, however, the same soap cannot be too mildly fitted, as it would liquefy at a temperature of over  $77^{\circ}$  F. if it did not contain sufficient soda or oils containing stearin. In cold weather the final fitting is carried out as much as possible with potash solution of  $28^{\circ}$  B. A sample dropped upon glass may show a strong "flower" and ring of lye, but, of course, fitting must not be carried too far. In summer the soap is only sufficiently fitted to insure its keeping ; a sample should show only slight "flower," and after cooling remain perfectly clear.

The proportion of soda is of importance as regards the durability of the soap, as well as the yield and the profit to be derived from it. While formerly the consistency of the soap was regulated by the use of softer or more solid fats and boiling with potash lye alone, at the present time the same oil is frequently used both in summer and winter, but the proportion of the soda to potash is changed. Both the solidity and the yield depend on the use of the soda, however, in an inverse ratio ; the more soda lye is used the more firm the soap becomes, but the smaller the yield. In midsummer about 50 pounds of soda lye of  $24^{\circ}$  B. are taken for every 100 pounds of oil, whereas in winter 25 pounds is the highest amount. In continued severe weather, and when working water-glass and oils or fats containing stearin in connection with the oil, soda lye is either entirely omitted or the proportion very much reduced.

When resin is added to the soap, as is generally done, 80 pounds of soda lye of  $24^{\circ}$  B. are allowed for every 100 pounds of it, because the soap would become too soft with the use of potash lye for the saponification of the resin.

Finally, we would remark that *the fabrication of all soft soaps is exactly the same as above described for the manufacture of soft soap from linseed-oil*, and that in order to obtain firm and durable products due consideration has to be given to the proportion of lime, content of moisture, fitting, and temperature.

*Glycerin soft soap.*—Under the name of "transparent glycerin soft soap," a product was some time ago introduced into commerce which differs from the ordinary soft soap in being lighter in

color. It contains, however, no other constituents which would justify the name given to it.

To obtain a product of a very light color the oil (linseed-oil or cotton-seed oil) intended for its preparation is generally first bleached. In working oil bleached with lye it must be taken into account that the oil becomes completely neutral by the removal of all fatty acids by the bleaching process, and that such neutral oil is difficult to combine with a large quantity of strong carbonated lye. In this it differs from unbleached linseed-oil, in the working of which the fourth part of the lye of  $18^{\circ}$  to  $20^{\circ}$  B. required can be at once added to the oil in the kettle. Bleached oil requiring a weaker and more caustic lye for combination, no more than one-fifth of the necessary lye can be brought into the kettle with such oil. A thorough union being established between the oil and lye, more strong lye is at once added to prevent the thickening of the soap.

To increase the yield and the lathering property of the soap, 5 to 15 per cent. of refined pale resin is generally added.

The lyes for this soap are prepared by slaking the lime (45 pounds for 100 pounds of 96 per cent. potash) in hot lye of  $6^{\circ}$  to  $8^{\circ}$  B. and dissolving in this the potash, with constant crutching. Of the lye obtained, that of  $15^{\circ}$  to  $17^{\circ}$  B. is used for combining the oil, and that of  $22^{\circ}$  to  $24^{\circ}$  B. for boiling, 100 pounds of linseed-oil requiring about 160 pounds of lye of  $24^{\circ}$  B.

The soap is generally prepared in the following manner: 1000 pounds of bleached linseed-oil, or, in the warmer season of the year, 700 pounds of bleached linseed-oil and 300 pounds of cotton-seed oil are brought into the kettle, together with about 400 pounds of lye of  $15^{\circ}$  to  $16^{\circ}$  B., and combined by crutching over a moderate fire. Combination being established, add quickly, in portions and with more vigorous boiling, lye of  $22^{\circ}$  to  $24^{\circ}$  B. until the soap forms a clear paste. In order to remove the superfluous water this paste is boiled until the scum disappears, the soap breaks audibly into roses, and when taken upon the spatula runs off in high streaks and breaks off short without spinning threads. As at this stage the moisture evaporates very quickly, notwithstanding the fire being kept very moderate towards the last, it is advisable to add now the quantity of lye



still wanting, to prevent scorching of non-saponified oil. Scorching is readily recognized by the odor and bluish vapors rising from the kettle.

When the soap shows the above signs of sufficient evaporation it is fitted to "flower" and "ring of lye." When suitably fitted, a sample, though spreading readily, should, after cooling, be clear, with a sharply-marked flower.

If, with a normal proportion of lime, the soap, after cooling, should show a transparent surface, but be turbid in the centre, lye is wanting, and lye of  $28^{\circ}$  B. must be carefully added until clearness is restored throughout the entire mass. If, however, the sample is whitish, turbid, short, and slippery on the glass, and does not become clear, or only so with great difficulty, fitting has been carried too far, and this defect is to be remedied by adding some linseed oil mixed with weak lye and allowing the whole to boil up.

When the soap boils up finished, and samples upon the glass are short, firm, and, after cooling, clear, 100 pounds of refined pale resin, together with about 92 pounds of lye of  $30^{\circ}$  B., are added, with vigorous stirring, and after allowing the whole to boil through, the soap is again fitted to "flower."

As the soap is rendered soft by the addition of resin, and pure oil soap boiled with potash lye alone in the warm season of the year lacks consistency, some soda lye of  $24^{\circ}$  B. is always used in boiling such soaps, the proportion varying from one-sixth to one-third, according to the season of the year and the amount of resin added.

The finished soap, after cooling to  $131^{\circ}$  to  $167^{\circ}$  F., is poured into barrels and stored in the cellar.

As regards the yield, it remains to be said that 100 pounds of bleached linseed and cotton-seed oil give about 230 to 233 pounds, and 100 pounds of resin about 140 to 150 pounds of well-boiled soap. The yield from unbleached linseed-oil is always somewhat less than from bleached.

We would finally mention a method of utilizing the dark precipitate resulting from bleaching linseed-oil with potash lye of  $28^{\circ}$  B. for soaps of a light color. Bring the precipitate into a kettle and boil it to a soft soap, with an excess of strong lye,

until it glides thin and without consistency from the glass; then segregate it almost completely by the further addition of lye. In this stage ladle the soap into a barrel and let it stand covered for a few days, when a clear, thick mass of soap, which, however, always remains soft, can be lifted from the dark lye. A portion of the soap-mass thus obtained can be used with other boilings, it being best to bring it into the kettle together with the oil, whereby combination is promoted. All dark oils, sediment of oil, and train-oil can be treated in the same manner, whereby they are not only rendered light in color, but almost odorless, so that they can be utilized as an addition in the fabrication of soft soaps.

*Soft soap from train-oil.*—Besides the above-mentioned smooth, transparent soft soaps, which, as previously mentioned, vary in color and the combination of fats, a variety boiled from train-oil with potash lye alone occurs in commerce, which is chiefly used in worsted spinning, as it gives to the yarn a specially brilliant color and possesses other good qualities, on account of which it is preferred to any other soap for the purpose.

*Soft soap from hemp-seed oil.*—This beautiful green soap is but little manufactured at the present time, and then chiefly for pharmaceutical purposes alone.

2. *Natural grain soft soap (fig soap).*—This is, no doubt, the best of all soft soaps. Its manufacture is, however, connected with difficulties, and the knowledge required for the preparation of a uniform and faultless product can only be acquired by considerable experience and close observation in boiling.

In the fabrication of this soap both hard fats containing stearin and softer fats and oils are used, the first yielding the "grain" and the latter the base-soap. Good materials only should be used, the tallow especially requiring to be as fresh as possible and free from dirt and acidity. Old tallow which has undergone a partial decomposition should be purified and prepared before use. This is done by melting and then heating to  $189.5^{\circ}$  F., and adding, with constant stirring,  $1\frac{1}{2}$  pounds of soda lye of  $38^{\circ}$  B., previously mixed with about 14 ounces of common salt for every 100 pounds of tallow. When the tallow has somewhat subsided add boiling water (6 pounds to 100 pounds of tallow), cover the kettle, and after standing a few hours ladle the

tallow off. Tallow prepared in this manner is well adapted for the manufacture of natural grain soap. If the soap-maker is, however, forced to use large quantities of old tallow without being able to purify it before use, it is advisable to work somewhat more carbonated lyes, any excess of carbonic acid being expelled in boiling the soap by the free oleic acid of the tallow. Horse-fat, lard, and bone-fat, which are frequently used, especially in summer, to make the soap more compact and able to resist the heat, must also be purified before use.

Other materials used are clear linseed-oil, or cameline-oil, and in summer one part of thoroughly refined cotton-seed oil. Saponified oleic acid, which otherwise is an excellent material in connection with other oils, especially for fulling soaps, is apt to make the soap dark, and must, therefore, be omitted where a light product is demanded.

The potash used in the fabrication of this soap should not contain more than two to three per cent. of soda, as otherwise the grain will be small and feather-like and the soap may even turn out entirely smooth. It is, therefore, advisable to procure from a reputable firm 96 to 98 per cent. potash guaranteed to contain only one-half per cent. of soda.

For the preparation of the lyes thoroughly burnt lime free from sand and grit is required. Slake the lime with hot lye of  $6^{\circ}$  to  $8^{\circ}$  B. and dissolve in this the required quantity of 96 per cent. potash. With good lime, and tolerably good water, 44 to 45 pounds of lime suffice in summer for 100 pounds of 96 per cent. potash and 40 to 42 pounds in winter.

A solution of 400 pounds of 96 per cent. potash is sufficient for 1000 pounds of fat. From 100 pounds of 96 per cent. potash 500 pounds of lye of  $24^{\circ}$  B. are on an average obtained, 155 to 160 pounds of which saponify 100 pounds of fat.

The first lye obtained from the solution is set at  $27^{\circ}$  to  $28^{\circ}$  B. and is used for boiling and filling, and the second, which is set at  $17^{\circ}$  to  $18^{\circ}$  B., for combining. Weaker lye than  $17^{\circ}$  B. is generally not used, because too much moisture would have to be evaporated, and, besides, lyes of  $12^{\circ}$  to  $15^{\circ}$  B. contain a considerable quantity of foreign salts in solution which in boiling act as carbonates and frequently exert even a stronger influence.



The lyes being prepared a determined quantity of fat is made ready for boiling. The composition of the fat varies very much, and depends on whether the soap is intended for the cold or warm season of the year, and the grain is to be small or large, or the color more pale or dark-yellow.

As regards the "grain" we would at once mention that its size in normally boiled soaps, *i. e.*, in soaps in which the content of water, the proportion of carbonic acid, and the fit are correct, depends entirely on the combination of fats chosen. The more fat rich in stearin is used in proportion to oil the more grain the soap will show, though it will be correspondingly denser and smaller because sufficient space for its development is lacking.

We submit a few formulas suitable for various conditions :—

1. *For pale yellow soap with rice-like grain. In summer:* Tallow 35 per cent., crude palm-oil 2, cotton-seed oil (or half lard) 33, linseed-oil 30.

*In winter:* Tallow 30 per cent., crude palm-oil 2, cotton-seed oil 33, linseed-oil, 35.

2. *For dark yellow soap with rice-like grain. In summer:* Tallow 35 per cent., crude palm-oil 3, cotton-seed oil or horse-fat 20, linseed-oil 42.

*In winter:* Tallow 35 per cent., crude palm-oil 3, linseed-oil 62.

3. *For pale soap with small rye-like grain. In summer:* Tallow 50 per cent., crude palm-oil 1, cotton-seed oil 15, pale linseed-oil 34.

*In winter:* Tallow 40 per cent., horse-fat or lard 9, pale linseed-oil 50, crude palm-oil 1.

4. *For dark soap with small rye-like grain. In summer:* Tallow 45 per cent., crude palm-oil 5, linseed-oil or cameline-oil 50.

*In winter:* Tallow 40 per cent., crude palm-oil 5, linseed-oil or cameline-oil 55.

5. *For soap with a beautiful medium grain. In summer:* Tallow 40 per cent., cotton-seed oil 25, pale linseed-oil or cameline-oil 35, crude palm-oil for coloring 1 to 2.

*In winter:* Tallow 40 per cent., linseed-oil or cameline-oil 60, crude palm-oil for coloring 1.

6. *For soaps for fulling purposes.* In summer: Tallow 34 per cent., palm-oil 6, oleic acid 40, linseed-oil 20.

In winter: Tallow 25 per cent., palm-oil 5, horse-fat or oleic acid 30, linseed-oil or cameline-oil 40.

Before entering upon the description of the process of boiling natural grain-soap, we would briefly mention the conditions which have to be closely observed in order to obtain a good product. Special attention must be paid to the proportion of lime, the content of moisture, and to fitting.

As mentioned in speaking of smooth transparent soaps, a salable soft soap cannot be prepared from pure caustic potash lye, as the result would be a gummy, viscid mass. To prevent this viscosity, and to give the product the required pliancy and salve-like consistency, a sufficient quantity of potassium-carbonate must be present in solution, which, by depositing itself between the atoms of soap, loosens the combination. This holds also good, but in a still greater degree, as regards natural-grain soap. In the preparation of this soap,  $\frac{1}{3}$  to  $\frac{1}{2}$  tallow is used, which always contains more or less free fatty acid, and for this reason such soap must contain a proportionally larger quantity of potassium salts in solution than smooth transparent soap, and the more so as on this depends the ability of the potassium stearate (grain) to move and develop, or, in other words, the possibility of crystallization and the formation of grain. Hence the more solid fat rich in stearin (tallow, etc.) in proportion to soft fat (oil, etc.) is used, the more carbonate the lyes employed for saponification must contain.

We have above spoken of too great causticity in the soap; we will now consider the opposite case. As already mentioned, the potassium salts, by depositing themselves between the atoms of soap, loosen the combination, and break up the viscosity. The more potassium salts are conveyed to the soap-mass, the looser and softer it naturally becomes, so that, with a further increase of potassium salts, the combination is finally destroyed, the potassium salts separating out, and, further, the soap being salted out. Hence, in order not to destroy the consistency of the soap-mass, there must be a determined proportion between the potassium salts and the caustic potash.

In order to carry on the boiling process with ease and rapidity, it is advisable to observe the following method of fabrication with the use of an average lye of  $23^{\circ}$  to  $24^{\circ}$  B. After once using a certain lye-pot for a boiling of soap, it will be known how many pots of lye of  $23^{\circ}$  to  $24^{\circ}$  B. are required for the saponification of, say, 200 pounds of fat. By noting the number of pots, the quantity of lye required for the next boiling can be quickly brought into the kettle, and evaporation of the soap proceeded with. Suppose 2000 pounds of fat are brought into the kettle, and 8 pots of lye of  $24^{\circ}$  B. are required for the saponification of 200 pounds of fat, then 34 pots full of lye of  $18^{\circ}$  B. are added to the fat in the kettle, and combination is promoted by crutching over a moderate fire. A thorough combination between fat and lye being established, 42 pots of lye of  $28^{\circ}$  B. are gradually added in portions of 10 to 12 pots with vigorous boiling, whereby paste will be formed. After thoroughly boiling the soap, about 5 pots of lye, which are still required, are gradually added, and, a sample being from time to time taken upon the glass, the sufficiently evaporated soap is finally fitted to a "slight flower."

The average lye is calculated in the following manner, according to the number of pots of lye of  $18^{\circ}$  and  $28^{\circ}$  B. which were required for the complete saponification of the fat:—

$$\begin{array}{rcl}
 34 \times 18^{\circ} & . & . & . & . & = 612 \\
 46 \times 28^{\circ} & . & . & . & . & = 1308 \\
 \hline
 80 \text{ divided into} & . & . & . & . & \frac{1920}{80} = 24
 \end{array}$$

Hence, by dividing 80 (the number of pots of lye used) into 1920, the figure 24 is obtained, which, in this instance, indicates the strength of the average lye.

We will now enter into the boiling of this soap, and the phenomena occurring thereon.

Combination being established with a slow fire, lye of  $28^{\circ}$  B. must be at once added to prevent the soap from becoming thick, and this addition is continued until paste is gradually formed. The soap is now allowed to boil up for the evaporation of the superfluous water. After adding nearly the entire quantity of the lye still required, and boiling for some time, the soap will



become thicker, clearer, and shorter. The scum upon the surface, which indicates superfluous particles of water, will gradually disappear, the soap commencing to boil in larger laminæ, and audibly "break into roses."

During the boiling, it must be observed whether the soap stands "correct in lime." Soap "too high in lime," even if thoroughly evaporated, is viscid and gummy after standing a few hours, and remains turbid and grains badly; soap "too low in lime" grains quickly, but shows a small and more round grain, and the transparent base-soap, lacking the required cohesion and consistency, becomes readily soft and syrupy.

Soap "too high in lime" boils heavily further down in the kettle, and rises only with a very strong fire. Further, the soap runs from the spatula in broad viscid streaks, and a sample dropped upon the glass will pile up high, congeal quickly, and be gummy and viscid. On the other hand, if the soap is "too low" in lime, it boils up high, rises over, is thinly-fluid, and samples dropped upon the glass spread out. Soap "correct in lime" boils easily and pleasantly, runs readily from the spatula in elevated streaks, lies for some time upon the glass, so that the rings formed can be observed until cooling: in breaking, the cooled sample is short like curd-soap. Hence, if natural grain-soap does not boil normally, solution of potash of  $28^{\circ}$  B. or caustic potash lye of  $23^{\circ}$  B. must be added according to requirement until it shows the requisite properties.

When the soap audibly "breaks into roses" and shows a correct proportion of lime, attention must be directed towards evaporation. A soap too strongly evaporated, and consequently deprived of the necessary content of water, grains very slowly and with difficulty, and sometimes not at all, on account of being too solid. If, on the other hand, the soap has sufficient moisture, the separation of the potassium stearate (grain) takes place with corresponding rapidity and regularity. In evaporating, the soap-boiler must principally be guided by the composition of the fats; the more solid fat, which fixes moisture, has been used the less the soap is to be evaporated. It is sufficiently evaporated when it boils in laminæ, audibly "breaks into roses," runs from the spatula in elevated streaks, and slips off from it short and with-

out spinning (drawing threads). Further, it should adhere in small drops to the finger dipped into a sample upon the glass.

The soap being sufficiently evaporated, it is adjusted to "flower." Samples upon the glass must show a "perceptible flower," be clear and firm after cooling, and after standing for some time appear no longer bright, but as if breathed upon. Special care must be had in fitting, for if the soap is too sharp the slighter crystals of palmitic acid contained in the fats, which are actually intended to make the soap more compact, would also incline towards the formation of grain, so that the soap would contain too much and wild grain, and, further, become slippery and separate lye. On the other hand, soap too slightly fitted, though forming a good grain, readily becomes soft and liquid. The soap is too sharp when a sample upon the glass runs over immediately and clarifies with difficulty or not at all; it is too weak when a sample upon the glass is not entirely clear after cooling, but shows a turbid point—the so-called "fat gray"—in the centre. Any necessary correction is made by adding fat, or lye of  $24^{\circ}$  B., as may be required. A sample upon the glass must show just sufficient "flower" to insure the durability of the soap.

If the soap is to be colored, the necessary quantity of crude palm-oil, together with the required quantity of lye, is added towards the end of boiling.

The finished soap being allowed to rest for some time, is slightly perfumed with oil of mirbane and then poured, at a temperature of about  $155^{\circ}$  F., into the barrels. The yield of unfilled natural grain-soap amounts to from 235 to 240 pounds from 100 pounds of fat.

The barrels containing the soap are then brought into the cellar, the temperature of which must be kept at from  $54.5^{\circ}$  to  $66^{\circ}$  F. in order to promote the formation of grain. At below  $54.5^{\circ}$  F. the crystals in the soap would congeal too quickly, and at over  $66^{\circ}$  F. remain in solution. The grain, which is a crystalline separation of potassium stearate and palmitate, forms in three to eight weeks, according to the proportion of tallow in the soap.

We would finally remark that in boiling natural grain-soap the temperature of the various seasons of the year must be taken into consideration, as, for instance, soap boiled in summer some-

times becomes turbid and wet by October. There is no one method of boiling natural grain-soap suitable for all cases, it being necessary to take into consideration the composition of the fat and the proportion of lime required for the respective season of the year.

*Natural grain-soap with stearin.*—In the same manner as tallow, pure stearin may also be used in the preparation of natural grain-soap, the grain obtained being especially firm and durable.

The process of fabrication is as follows: 1200 pounds of linseed or cameline-oil (in summer one-third cotton-seed oil) are brought into the kettle, together with about 500 pounds of lye of  $20^{\circ}$  B., and combined over a moderate fire. Combination being established, lye is quickly added to prevent thickening of the mass. The fire being increased, more lye of  $20^{\circ}$  B. is introduced in portions until paste is formed, which is thoroughly boiled, with the successive addition of the quantity of lye of  $28^{\circ}$  B. still required, until the superfluous water is evaporated. When no more scum appears upon the surface, and the soap boils in laminae and audibly “breaks into roses,” and when thrown shows no more water-bubbles, but runs in elevated streaks from the spatula and slides off short, fitting is proceeded with. With a correct fit the samples upon the glass must be clear, firm, and short, show a good “flower,” and remain clear after cooling. To the soap boiling up finished add 100 pounds of stearin, together with about 125 pounds of potash solution of  $40^{\circ}$  B., and allow the whole to combine. A sample upon the glass must again show a “good flower” and remain clear after cooling.

The finished soap, being cooled to about  $156^{\circ}$  F., is poured into barrels and the latter brought into the cellar, where, with a suitable temperature, the grain will soon form.

3. *Artificial grain-soap.*—This soap occurs in commerce of a yellow, green, or brown color, and is prepared by saponifying linseed-oil, cameline-oil, cotton-seed oil, sesame-oil, sulphur olive-oil, and peanut-oil, and sometimes oleic acid with potash, and eventually soda lye of  $20^{\circ}$  to  $25^{\circ}$  B. Generally 10 to 15 per cent. of dark or pale resin is used in connection with the oils. The artificial grain for this soap is chiefly prepared from chalk or thoroughly burnt lime. The chalk-grain, various sizes of



which are found in commerce, being hard and dissolving with difficulty, sinks to the bottom of the wash-tub in using the soap and is, therefore, very much disliked. Lime-grain, however, shows none of the above disagreeable properties, but as it cannot be had in commerce the soap-boiler has to prepare it himself. For this purpose well-burnt lime is pounded, passed through a medium-meshed wire sieve, and then freed from lime-dust by means of a hair-sieve. The small irregular pieces remaining in the hair-sieve form the "grain," and are preserved for future use in a large well-stoppered glass-balloon. Later on  $3\frac{1}{2}$  to 14 ounces of them are added to about 200 pounds of the somewhat cooled soap. The lime soon slaking in the warm, liquid soap expands and forms a grain similar to the natural one.

*Yellow artificial grain-soap.*—This is a golden-yellow, transparent soap, which is chiefly prepared from refined cotton-seed oil and bleached linseed-oil and potash lye, some crude palm-oil being generally used for coloring.

The soap is prepared in a similar manner as given for smooth, transparent soft soap. Combine 1000 pounds of oil with lye of  $15^{\circ}$  to  $16^{\circ}$  B. over a moderate fire. Combination being established, add quickly more lye of  $20^{\circ}$  to  $24^{\circ}$  in portions of 200 to 300 pounds, allowing one portion to combine before adding the next, until a clear paste is formed. The fire being increased, allow the mass to boil high, whereby the scum will gradually disappear and the paste improve in clearness and consistency. By continued boiling the soap will soon be freed from superfluous water and scum, boil in large laminae, audibly "break into roses," and, when thrown with the spatula, break off short and have a woolly appearance. At this stage the palm-oil required for coloring is added to the gently boiling soap and the latter then fitted to "flower." With a correct fit samples upon the glass must show a "perceptible flower," and after cooling be clear, short, and firm.

If the samples are transparent on the surface but turbid in the centre, the soap is too weak, and lye is to be carefully added until it shows sufficient "flower" and remains clear on cooling. If, however, the sample runs over quickly, is turbid after cooling, and short and slippery upon the glass, the soap is too sharp, and

oil mixed with weak lye is to be added until a sample remains entirely clear after cooling.

Crutch into the finished soap, cooled to about  $167^{\circ}$  F., 5 to 6 pounds of artificial grain and pour into dry barrels.

*Green artificial grain-soap.*—Formerly this soap was prepared from hemp-seed oil, but as the supply of this oil decreased, linseed-oil was substituted and the resulting yellow soap colored green chiefly with indigo prepared in the following manner: For 1000 pounds of oil from 8 to 10 ounces of good indigo were rubbed fine and boiled with lye of  $23^{\circ}$  to  $24^{\circ}$  B. until the whole had acquired a soapy consistency. A drop of this boiled indigo added to a glass full of rain or river water had to produce a green coloration; if the water acquired, however, a more bluish-green color, some more lye was added to the indigo and the whole boiled for some time longer. The indigo thus prepared was diluted with some lye and added to the finished soap, giving it a beautiful green color.

In modern times the soap is generally colored with ultramarine blue mixed with water or lye, 2000 pounds of oil requiring about  $1\frac{1}{2}$  to 3 pounds of ultramarine for prime green soap.

In winter linseed and cameline-oils alone are used for the preparation of green soap; in summer a part of these oils is, however, replaced by cotton-seed oil, rape-oil, and sesame oil, as well as by horse-fat and train-oil. In winter the soap is generally boiled with potash lye alone, but in summer 20 to 30 per cent. of soda lye are used to give the soap a better consistency.

The lyes are prepared in the same manner as for other soft soaps. The lime is slaked in hot lye of  $7^{\circ}$  to  $8^{\circ}$  B. and the potash dissolved in the resulting milk of lime. Since 100 pounds of 96 per cent. potash saponify about 270 pounds of oil, about 400 pounds of 96 per cent. potash are required for 1000 pounds of oil, and about 170 pounds for causticizing the potassium carbonate. However, in order to have some lye in reserve in case of emergency, it is advisable to set 500 pounds of 96 per cent. potash and 215 pounds of lime. The soap is boiled with an average lye of  $23^{\circ}$  to  $24^{\circ}$  B., about 160 to 165 pounds being required for the saponification of 100 pounds of oil.

The manner of preparing green soap is as follows: 1000

pounds of oil, together with 400 pounds of lye of  $18^{\circ}$  to  $20^{\circ}$  B., are brought into the kettle and allowed to stand over night, whereby a kind of emulsion is formed. The next morning the oil and lye are combined over a moderate fire, and as soon as combination is established more lye is added. By careful firing and adding lye at the proper time any thickening of the mass is prevented. After gradually adding more lye of  $27^{\circ}$  to  $28^{\circ}$  B., and increasing the fire, paste is formed, which will become clearer and of a better consistency by the addition of more lye and continued boiling. The required lye having been gradually added, the fire is increased and the soap thoroughly boiled to remove the superfluous water. With a kettle wide on the top, which offers the clear boiling soap, a large surface for evaporation, fabrication proceeds rapidly, the scum will soon disappear, and the soap boil in laminae and commence to "talk." The soap is finally fitted to a "good flower," so that a cooled sample is clear and firm and does not become turbid after standing for some time.

If resin is to be added to the soap, about 100 pounds of comminuted pale resin, together with 92 pounds of lye of  $30^{\circ}$  B., are added to the finished soap and the whole boiled through. The soap is then once more carefully fitted. The addition of resin rendering the soap soft, it is necessary to use one-fifth to one-third of soda lye of  $24^{\circ}$  B., according to the season of the year and the amount of resin.

The finished soap being colored in the manner previously described, it is allowed to rest for some time. When cooled to about  $155^{\circ}$  to  $167^{\circ}$  F., the lime-grain (1 pound to 1000 pounds of oil) is crutched in, the soap perfumed with some oil of mirbane, and then poured into dry barrels.

*Brown artificial grain-soap* is prepared by boiling dark resin and some wool-fat with the oil, or coloring the finished soap with sugar color.

4. *Soft soap with a mother-of-pearl lustre.*—This soap, also known as elaëidin soap, silver soap, etc., is much liked for household purposes, as well as in the textile industries. In its fabrication various combinations of fats are used, consisting of tallow, horse-fat, lard, bone-fat, oleic acid, palm-oil, linseed-oil, cameline-oil, peanut-oil, and cotton-seed oil, the latter being much used at



the present time on account of its low price. Palm-oil is also much worked in a crude state for coloring, which imparts to the soap a yellowish appearance, as well as in a bleached state, by which the soap acquires an agreeable odor of orris root.

The lyes required for the fabrication of this soap are prepared in the same manner as given for other soft soaps. Potash lye of  $20^{\circ}$  to  $25^{\circ}$  B. is generally used for boiling in connection with one-third to one-sixth of soda lye of  $25^{\circ}$  B., according to the season of the year and the amount of filling. In working large quantities of solid fats a corresponding reduction of the proportion of soda is required. The radiated silvery appearance of the soap is produced by the soda lye, the sodium salts combining with the particles of stearin and palmitin in the fats to white silvery streaks. The soap acquires the silvery appearance only after complete cooling and storing for 8 to 14 days.

A suitable combination of fats has to be chosen for the preparation of this soap, according to the prevailing temperature and season of the year. We submit a few suitable formulas:—

*For white elaidin soap. In summer:* I. Cotton-seed oil 70 pounds, tallow 25, bleached palm-oil 5.

II. Cotton-seed oil 75 pounds, tallow 25.

III. Cotton-seed oil 60 pounds, lard 35, bleached palm-oil 5.

IV. Cotton-seed oil 60 pounds, tallow 20, lard 20.

*In winter:* I. Cotton-seed oil 75 pounds, lard 20, bleached palm-oil 5.

II. Cotton-seed oil 80 pounds, lard 20.

III. Cotton-seed oil 60 pounds, peanut-oil 20, lard 20.

IV. Cotton-seed oil 75 pounds, lard 15, bleached palm-oil 10.

*For yellow soap. In summer:* I. Cotton-seed oil 60 pounds, tallow 35, crude palm-oil 5.

II. Cotton-seed oil 60 pounds, horse-fat\* 35, crude palm-oil 5.

*In winter:* I. Cotton-seed oil 75 pounds, tallow 20, crude palm-oil 5.

II. Cotton-seed oil 60 pounds, linseed-oil 25, tallow 15.

*For good fulling soap. In summer:* Horse-fat 50 pounds, oleic acid 10, linseed-oil 40.

\* By horse-fat is understood a mixture of horse-fat, tallow, and lard as furnished by establishments where dead animals are boiled.

*In winter:* Linseed-oil 50 pounds, oleic acid 10, horse-fat 40.

In preparing these soaps the proportion of lime first deserves attention. To produce a beautiful, delicate, and white soap more strongly carbonated lyes are required for boiling, as a soap "too high" in lime would turn out too solid and dark, the proportion of carbonate to caustic potash in the lyes should be about as 1 : 2. As the soap-boiler does not like to change the proportion of lye to which he is accustomed, he can boil with his ordinary lye and bring the soap to the required proportion of lime by the addition of potash solution of  $24^{\circ}$  B., and in this manner can use as much as 25 pounds of potash solution of  $24^{\circ}$  B. for 100 pounds of fat, according to whether the lyes are more or less high in lime.

The addition of soda lye varies according to the composition of the fats and the season of the year, less being required the more tallow is used, while for soaps chiefly prepared from cotton-seed oil, lard, and horse-fat, as much as 33 pounds of soda lye of  $25^{\circ}$  B. may be worked for 100 pounds of fat.

Special attention must also be paid to fitting. The soap should only show a "slightly perceptible flower" and become white only after some time. If a sample becomes white at once and glides upon the glass, some fat has to be added, as the soap, being too sharp, would later on become short and separate lye. On the other hand, the fit must not be too slight, as the soap would become soft and "long" on storing.

The soap is generally only evaporated until it "breaks into roses." In this stage it is fitted, which consists chiefly in shortening it, and can be executed by scattering calcined soda over it.

*Smooth elaidin soap.*—This soap can be prepared in winter from cotton-seed oil; in summer, however, it is necessary to use in connection with the oil more solid or semi-solid fats, together with more soda lye, as otherwise the soap would be too clear and lose its silvery appearance.

The process of boiling is generally as follows: 1000 pounds of refined cotton-seed oil and about 400 pounds of potash lye of  $16^{\circ}$  to  $17^{\circ}$  B. are brought into the kettle and combined over a moderate fire. Combination being established add 500 pounds of lye of  $18^{\circ}$  B. to the mass and increase the fire. Then with constant boiling and vigorous firing, add 600 pounds of potash

lye of  $24^{\circ}$  B. in portions of 200 to 300 pounds each and 250 pounds of soda lye, whereby a clear paste will be formed, which is freed from superfluous water by thorough boiling. The mass now becomes thicker, clearer, and shorter, the scum gradually disappears, and the soap boils in laminæ, runs from the spatula in elevated streaks without spinning threads or breaking off, and audibly "breaks into roses." During the boiling 40 to 50 pounds of potash solution of  $24^{\circ}$  B. are generally added which renders the soap clearer and more pliant, the soap being also fitted as much as possible with potash solution while it "breaks into roses." With a correct fit a sample upon the glass must show "flower" and otherwise be clear, sufficiently short, and firm. The sample being thoroughly cooled, a white ring with thread-like, radiated crystallization extending towards the centre must first show itself in the still transparent soap. At the same time a star-like crystallization forms in the centre, which extending outward soon grows through the entire sample and gives it the lustrous, mother-of-pearl appearance. The same process of crystallization takes place later on in the barrels.

If the sample upon the glass becomes, however, white at once and slippery, the soap is too strong, and oil must be added until the sample shows a "slighter flower" and becomes white and lustrous only after cooling. If, on the other hand, the sample shows only a slight sign of "flower" and does not keep clear until cold, but is whitish turbid in the centre, the soap is too weak, which is remedied by carefully adding lye of  $25^{\circ}$  B. and eventually potash solution.

When the fitted soap "breaks into roses" and appears sufficiently short, evaporation is discontinued and the fire removed. The soap remains standing overnight in the covered kettle. The next morning the scum formed upon the surface is removed and large samples are laid aside to cool. The behavior of these samples indicates whether the soap is good in all respects or whether it can be improved by crutching in strong potash lye or potash solution. Such corrections must, however, be done very carefully, as with an addition of too strong potash solution the soap becomes too white and with potash lye too firm.

After perfuming the finished soap with oil of mirbane, which



somewhat covers its not very agreeable odor, it is poured into barrels.

The use of peanut-oil is still more advantageous for the preparation of this soap than that of cotton-seed oil, as the soap acquires the white, mother-of-pearl appearance with a small addition of soda lye, and is also more pliant and whiter than that from cotton-seed oil.

*White soft soap.*—Under the name of *bleaching soap* or *white soft soap* a product is brought into commerce which is a soft soap only in name and consistency. It is generally prepared by boiling 100 pounds of cocoanut-oil to a paste quite free from scum, with about 200 pounds of soda and potash lye of  $20^{\circ}$  to  $21^{\circ}$  B., fitting strongly and crutching into the paste 500 to 600 pounds of hot soda water-glass previously mixed with 50 to 60 pounds of lye of  $20^{\circ}$  B. The mass is perfumed with oil of mirbane or lavender oil, and finally stirred until cold. Palm-kernel oil and bleached palm-oil are frequently substituted for a portion of the cocoanut-oil.

In Southern Germany a so-called tallow soft soap is prepared by heating 100 pounds of tallow with 250 pounds of caustic soda lye of  $3^{\circ}$  B. over a moderate fire until a good combination is established, then adding successively 250 pounds of caustic soda lye of  $7^{\circ}$  B. and heating the mass, without, however, allowing it to boil, until it is quite clear. The soap, after resting for some time, is perfumed with oil of mirbane and poured into barrels.

*Filling of soft soaps.*—For the purpose of artificially increasing the yield of soft soap it is first of all necessary that it should have been correctly boiled and fitted, *i. e.*, that a complete saponification of the fats by the alkalies has taken place. Further, the soap to be filled must be sufficiently evaporated, so that it can better absorb and hold the filling material. And as most filling materials possess shortening properties, the soap must also stand "well in lime," *i. e.*, somewhat more caustic lye must be used in boiling. Only a compact, healthy base-soap (with not too high a content of resin) is capable of absorbing a suitable filling material.

As the soap-boiler can never know in advance the quantity of filling material the soap may be able to absorb without suffering

in consistency and appearance, it is advisable to determine the amount by a test with a small quantity of finished soap. The soap is generally filled, when cooled, to about  $167^{\circ}$  to  $178^{\circ}$  F., the materials chiefly used at the present time being potassium chloride, "gelatine," water-glass, "sapolite," potash filling, filling lye, and, where the appearance of the soap permits, potato-flour. The smooth, transparent, and artificial grain soaps being the ones chiefly filled, we give as follows, the filling materials used and their behavior :—

For filling these soaps, potassium chloride is perhaps used more than any other material. Shortly before use, it is dissolved in water, and the solution set at  $13^{\circ}$  to  $14^{\circ}$  B. Of this solution, 20 to 25 per cent. of the fat used is crutched into the finished soap, cooled to about  $167^{\circ}$  F., and the latter is again fitted with strong caustic lye. Soaps filled with solution of potassium chloride are capable of strongly resisting the cold in winter, and, with a very much reduced proportion of soda, do not need too strong a fit.

"Gelatine," brought into commerce by several manufacturers, is also much used for filling these soaps, 20 to 25 per cent. being crutched into the finished product, without a further fitting being required. As the "gelatine" is prepared chiefly from soda water-glass, it is advisable to make a corresponding reduction in the proportion of resin and soda.

The so-called "sapolite," brought into commerce by a firm in Offenbach, Germany, is nothing but a mixture of potassium chloride and sodium sulphate. It is used in the following manner: Dissolve 1 part of sapolite in 2 parts of hot potash lye of  $6^{\circ}$  B., and add  $2\frac{1}{2}$  to 3 parts of water. The resulting clear solution should show about  $16^{\circ}$  B. (in winter  $20^{\circ}$  to  $22^{\circ}$ ). About 20 to 25 per cent. of the oil used of the solution is then crutched into the cooled soap. Soap thus filled has a beautiful, clear appearance, but as a sample upon the glass must show a vigorous "flower," subsequent fitting is sometimes required. A reduction of the proportions of resin and soda is also recommended with the use of this filling material.

There are numerous receipts for the preparation of the so-called filling lye, of which we give two very good ones :—

I. Saponify 50 pounds of cocoanut-oil with 100 pounds of

soda lye of  $20^{\circ}$  B., and crutch into the resulting soap 200 pounds of potassium chloride and 100 pounds of calcined soda dissolved in 2000 pounds of water.

II. Dissolve 100 pounds of half-grain or grain soap in 600 pounds of hot water, and crutch into the solution 100 pounds of solution of potassium chloride of  $15^{\circ}$  B. and 150 pounds of soda water-glass of  $38^{\circ}$  B.

About 20 per cent. of the cold filling-lye can be added to the cooled soap, which must, however, be again fitted.

Potato starch is largely used for filling soft soaps. It does not swell much either in cold water or in an alkaline solution, but when caustic lye is added, it is at once converted into a smooth, stiff jelly. It has further the property of fixing much moisture, thereby rendering the filled soap more compact and durable. As, however, soap filled with potato starch is more turbid, only the poorer qualities are filled with it.

Filling with potato starch is done in the following manner:—A large vat of soft wood, for the reception of the filling material, is placed near the kettle. Pour into the vat 1 part of water or potash solution of  $5^{\circ}$  to  $8^{\circ}$  B., stir in 1 part of potato starch, and finally crutch in 1 part of soda water-glass. The latter may also be omitted, using instead 1 part of potato starch in 2 parts of solution of potash, or of potassium chloride of  $10^{\circ}$  to  $12^{\circ}$  B. The potato starch being thoroughly stirred in, introduce sufficient finished soap from the kettle to form a cream-like mass, which is added to the soap in the kettle and thoroughly crutched in. The soap, becoming soft, viscid, and “long” by the addition of the filling, must be again fitted with caustic lye of  $27^{\circ}$  to  $30^{\circ}$  B., which is added with constant crutching, until a sample upon the glass is again full and round and firm, and shows the necessary sharpness.  $1\frac{1}{2}$  parts of fitting lye of  $27^{\circ}$  B. are generally allowed for 1 part of potato starch.

Natural grain soap is also somewhat filled at the present time, the principal materials used being prepared potash water-glass, potassium filling,\* and potato starch. In using the latter, the soap must show a good content of moisture.

\* Prepared potassium water-glass and potash filling are manufactured by Messrs. van Baërle & Sponnagel, of Berlin, Germany.



## CHAPTER XVII.

## SOAPS FOR THE TEXTILE INDUSTRIES.

THE soaps used in the textile industries may be divided into bar-soaps and barrel-soaps. The bar-soaps are either boiled clear upon sub-lyes, or they are grain-soaps prepared upon a precipitate of paste. The barrel-soaps are boiled like natural grain-soaps and are prepared either with pure potash lye or with the partial use of soda lye; they are all strongly alkaline and are chiefly used for medium and ordinary woollen goods.

There is quite a demand for natural grain-soaps with a clear ground and large, firm grain, such soaps offering a guarantee to the consumer that only pure potash lyes have been used in their preparation. Those accustomed to these soaps do not like to give them up, as the strong alkaline solutions possess the necessary power of washing and felting the goods. Where, however, soaps with a torn grain—which is produced by a small addition of soda lye—have been introduced, soaps with a hard grain are not liked, because they do not dissolve as readily as the former. This kind of soap gives a yield of 220 to 250 per cent. Soaps with an abundance of small grain, prepared from pure oleic acid, are also much used, good saponified oleic acid with a suitable addition of tallow-like fats being required for their preparation. To this class belong also the smooth white soaps boiled from mixed fats, oleic acid, cotton-seed oil, sesame-oil, peanut-oil, and perhaps some bleached linseed-oil, with the use of one-third soda. These soaps give a yield of 220 to 230 per cent.

Further, there are soft soaps which are boiled from oils, such as linseed-oil, rape-oil, distilled oleic acid, etc., and are used for fulling goods of a poorer quality, and finally, very soft potash soaps from cameline-oil, which are exclusively used in spinning in place of oil for moistening the thread.

Under the name of "economy soap," another soap has been recently introduced which excels in its high content of fatty acid, being superior in this respect to natural grain-soap.

For fulling every good soap may be used in which the fats are thoroughly saponified, and which on dissolving gives a dense paste uniformly spinning threads from above to below, and in cooling congeals to a homogeneous mass, and besides possesses the required detergent power and the property of promoting the felting of the woollen goods. In this respect soaps from tallow-like fats, such as palm-oil and olive-oil, boiled either as curd-soaps without a material addition of cocoanut-oil or palm-kernel oil, or as soft soaps without a large quantity of linseed-oil, rape-oil, or hemp-oil, have stood the test.

It is advisable for the soap-boiler who wishes to furnish fulling soaps not to imitate a soap already introduced after making a superficial examination, but to communicate with the consumer and ascertain the content of fatty acid and alkali which is demanded.

Whether soaps are serviceable for the purpose can be readily determined. If only tallow-like fats have been used, dissolve about  $3\frac{1}{2}$  ounces of soap in about 2 pounds of boiling water and allow the solution to cool. The saponification is defective if a curd soap-like coating is separated or the soap does not congeal uniformly from above to below, is not clear, adheres to the glass, or settles as an aqueous solution to the bottom; further, the cooled solution must not undergo a change by the addition of 4 to 6 per cent. of crystallized soda and 1 to  $1\frac{1}{2}$  per cent. of common salt. If fats have been used yielding soaps which do not solidify after cooling the solution, the latter should be correspondingly clear and not separate a curd soap-like coating.

### *Bar Soaps.*

*Ordinary fulling soaps.*—Caustic lye is used for boiling where there is no objection to the soaps containing free alkali and having a sharp touch. First of all the soap-boiler should, however, find out what kind of goods are to be fulled, as sharp soaps can only be used for fulling ordinary woollen goods of a natural color,

they attacking every dyed stuff. Such soaps are but seldom demanded.

If, on the other hand, the soap-maker wishes to compete with a soap already introduced, it becomes necessary to imitate it as closely as possible. For this purpose procure a sample of 20 pounds or more and compare it with the imitation. It is also advisable accurately to test the sample as to its content of fatty acid and alkali. Though this is not of great assistance in the manufacture of the soap itself, it is in so far useful as by the analysis it can be determined whether the soap has been correctly imitated.

The following example may serve as a guide for all such cases. Suppose in 100 parts by weight of soap were found 60 parts of fatty acids, 6.25 parts of soda, and 30 to 35 parts of water. This would be one of the best soaps for the textile industries. To prepare such soap there would be required about 65 parts by weight of fat, 14.5 parts of 70 per cent. caustic soda, 5 parts of salt, and as much water as needed. If the chemist finds in 100 parts by weight of soap 62 parts of fatty acids, 7.25 parts of soda, and 29 to 33 parts of water, he says the soap contains much alkali; it is not neutral. It is a clear boiled soap like many household soaps. Such soap could be prepared from about 68 parts by weight of fat, 15 parts of 70 per cent. caustic soda, 3 parts of salt, and as much water as required. If 63 parts by weight of fatty acids, 8 to 9 parts of soda, and 27 parts of water are found in a curd-soap, it may be assumed that the soda has been introduced in excess either at once in boiling or subsequently. Such soap would be prepared from 69 parts of fat, 15 parts of 70 per cent. caustic soda, 2 parts of salt, and as much water as required. The clear soap, thoroughly evaporated, might have eventually to be ground with potassium carbonate and brought into low frames. For imitating the last soap the following formulas might be used:—

I. Tallow 1000 pounds, bone-fat 500, oleic acid 300, peanut-oil 200, 70 per cent. caustic soda 425, salt 60.

II. Tallow or palm-oil 1000 pounds, cotton-seed oil, 500, saponified oleic acid 300, peanut-oil 200, 70 per cent. caustic soda 425, salt 60.

III. Tallow or palm-oil 500 pounds, bone-fat 500, horse-fat



500, distilled oleic acid 400, palm-kernel oil 100, 70 per cent. caustic soda 425, salt 60.

The process of boiling is executed by first dissolving in the kettle the caustic soda to a lye of  $15^{\circ}$  B. A portion of the lye is then ladled into a receptacle standing near the kettle, and the oleic acid first added to the boiling lye remaining in the kettle. Saponification having taken place, the palm-kernel oil, horse-fat, bone-fat, etc., are brought into the kettle, the soft fats being, as a rule, introduced first and the solid fats last. If a thoroughly combined soap be not formed after the introduction of all the fats, or the soap is thick, water is poured over it until it boils regularly, after which the remaining lye is added. Boiling is now continued until a soap free from froth is formed, which, though it may "wet" considerably, becomes solid after cooling. Now evaporate so far that the lye-power commences to exert a disintegrating effect, as clear boiling will then not be necessary after adding the salt. But little experience is required to manage so that after the addition of the salt the soap lies a bright grain in the kettle, it being effected by moderating the steam or the fire so that actual boiling no longer takes place. The salt converted with water into a solution showing  $23^{\circ}$  B. is then sprinkled over the soap and the latter not allowed to boil up until all the solution is in the kettle. The soap then boils up clear and boiling is continued with the kettle at first half covered and finally entirely. Such soap forms quite a thick grain, samples of which are allowed to cool and then tested as to the characteristics such soap should show.

If the soap is not clear after adding the salt, boiling must, of course, be continued until by the evaporation of more water such is the case. Everything else to be done with such soaps is a matter of experience. Some water, weak lye, or carbonated solutions might be sprinkled over very thick soap, and this be continued until by the appearance and taste of samples repeatedly taken it is seen that the soap possesses the desired properties. If the soap is to be smooth, it is brought into low frames; if marbled, into large ones.

For the above process of boiling in the direct way, the fats as well as the lye used should be as free from dirt as possible, as

otherwise the greater portion of the dirt remains adhering to the soap.

These soaps can also be obtained as pure and of as light a color as possible by repeatedly boiling the grain upon fresh weak lye. The clear, thick grain will then have to be ground with water, constant ebullition being kept up until it is entirely liquefied and the lye begins to run off pasty. After removing the sub-lye, weak lye of  $3^{\circ}$  to  $4^{\circ}$  B. is introduced and perhaps more salt water, this being repeated as often as required. The purified grain is then tested as to its properties.

*Neutral curd-soaps.*—These soaps are more frequently used in the textile industries than the preceding varieties. They can only be prepared by the use of suitable fats. It may sometimes succeed to prepare them from tallowy or lardaceous fats as well as from oleic acid, cotton-seed oil, peanut-oil, etc. They being, however, frequently defective after the thorough grinding with water which is required, lye has to be again added in order to obtain a product of good appearance, so that the resulting soap will no longer be entirely neutral. The only oil suitable for the preparation of fine, neutral soap is olive-oil. If this is thoroughly boiled and the soap correctly treated afterwards, the result will be a lean, compact, neutral soap, which will not injure the most sensitive color upon cotton or silk, but even increase its lustre.

The following may serve as a formula for as neutral a tallow or palm-oil soap as possible: Tallow or palm-oil 1200 pounds, lard or horse-fat 500, peanut-oil 300, 70 per cent. caustic soda 400, common salt 120.

Three different kinds of lye are prepared from the caustic soda, one-third of it being dissolved to lye of  $10^{\circ}$  B., one-third to  $15^{\circ}$  B., and one-third to  $20^{\circ}$  B. The salt is also converted with hot water to a solution of  $23^{\circ}$  B. All the fats are brought into the kettle, together with lye of  $10^{\circ}$  B. (30 pounds to 100 pounds of fat), and heated to boiling. Combination being established, boiling is continued with the remaining lye of  $10^{\circ}$  B. and then with the lye of  $15^{\circ}$  B. Finally the lye of  $20^{\circ}$  B. is gradually added, the effect produced by each portion being awaited before adding the next. If the soap should not absorb the last portion, but tear

apart like curd-soap, water is added until absorption is effected. The paste-soap is then allowed to boil for several hours until it is perfectly clear and free from scum. Separation is then effected by pouring one pat of salt water after the other over the quiet soap, shutting off the steam or fire in the meanwhile.

All the salt water having been added, and allowed to act for some time, steam is again admitted, or the fire again started, the soap, as a rule, boiling up free from scum, though even if such be not the case, it is of no importance as long as the soap rises up bright.

If the grain in the kettle is very thick, water is gradually poured over it, the whole being kept in constant ebullition, and the distribution of the water assisted with the crutch or spatula, until the soap liquefies more and more, and boils high. It is then tested as to firmness and touch. If touch is found to exist, some lye is drawn off or pumped out, the mass allowed to boil thoroughly through, and water poured over it in the same manner as above. This is continued until the result is a lean, solid soap, showing no touch of lye. During the entire operation, which is rather a washing out than boiling of the grain, the soap must be managed so as to keep bright and clear.

The process of grinding and consequent washing out of adhering alkali can, however, be only carried to within a certain limit, the soap otherwise becoming dull and frequently entirely frothy. Such dull soap pastes, and compares unfavorably with olive-oil soap, and cannot be restored, like the latter, by the addition of salt water, but only with lye, and hence soaps for the textile industries prepared from tallowy fats are only approximately neutral. The finished soap is brought into large frames, where much of the lye still adhering to it separates out.

The following formulas can be subjected to the same treatment as above: I. Tallow or palm oil 800 pounds, bone fat 600, peanut-oil and saponified oleic acid, each 300, 70 per cent. caustic soda 400, salt 120.

II. Tallow or palm oil 600 pounds, bone fat 600, saponified oleic acid and peanut-oil and 70 per cent. caustic soda, each 400, common salt 120.

The caustic soda lye or a portion of it can be replaced by tank



lye of  $15^{\circ}$  B., 200 pounds of it being allowed for 100 pounds of fat.

Olein soaps are also much used in the textile industries, but the final result of the boiling process being similar to that of the preceding, they turn out still less neutral, as, on account of cooling with difficulty, they have to be either crutched cold in large frames, or brought into small frames, which prevents the adhering lye from thoroughly separating out. However, a small addition of hard, tallowy fat is always used for these soaps. We submit the following formula:—

Saponified oleic acid 1600 pounds, tallow or palm oil and 70 per cent. caustic soda, each 400 pounds, salt 120 pounds.

Dissolve in the kettle the 400 pounds of caustic soda to a lye of  $16^{\circ}$  B., and gradually add the oleic acid, allowing one portion to combine before adding the other. The greater portion of the oleic acid being in the kettle, introduce one-half of the common salt, and finally the tallow or palm-oil. This soap must also boil for some time, portions of the sub-lye being from time to time removed and replaced by water. A strongly evaporated curd-soap boiling finally upon scant lye, solution of carbonate of  $3^{\circ}$  to  $4^{\circ}$  B. is poured over it until the soap again rises, the desired object being thus attained.

A nearly neutral soap results from the following formula:—

Tallow 600 pounds, olive-oil 1400, 70 per cent. caustic soda 400, common salt 120.

The various brands of olive-oil offered to the soap-boiler may vary very much. One lot may be old oil no longer suitable for table purposes. This is thinly-fluid, and, if worked by itself, would not yield a solid bar-soap, so that a greater percentage of tallow than given in the formula would be required, either half and half or 12 parts tallow and 8 parts oil. Such thinly-fluid oil is also found among the so-called fabricated oils (*huile d'olive à fabrique*), which also require an addition of hard fats. The very thick sedimentary oils and also the green oils, so-called sulphur oils, are generally boiled without an addition of other fats. The latter, however, when bleached, would be very suitable for the above formula in the proportion given.

The olive-oil is boiled in a similar manner as oleic acid, the

caustic soda being dissolved in the kettle, and the lye set at  $20^{\circ}$  B. The oil is then added to the moderately boiling lye and finally the tallow. At this stage, boiling is continued until the fats are thoroughly saponified, when the dissolved salt is added.

To a soap-boiler used to quick work, or having little time, the preparation of these olive-oil soaps is a very tedious process, the boiling and grinding from the time the salt water is added for separation requiring fully twenty-four hours. For this reason, boiling with indirect steam is almost absolutely necessary, as well as an arrangement for drawing off or pumping out the salt-lye. The process of grinding the soap resulting from the above combination being almost the same as for pure olive-oil soaps, we refer to the following:—

*Soaps from green sulphur olive-oil.*—For pure olive-oil soaps, the thick sedimentary oils or green sulphur oils alone are used. The following may serve as formulas:—

I. Sulphur olive-oil 2000 pounds, caustic soda 425, common salt 200.

II. Sedimentary oil or bleached sulphur oil 2000 pounds, caustic soda 400, common salt 80.

The boiling process is the same for both formulas, only some more alkali being used for sulphur oil on account of the color, the resulting soap requiring also some more salt. The preparation of the lyes for the two oils also differs somewhat. In both cases the soda is dissolved in the kettle, the resulting lyes being, however, set at  $25^{\circ}$  to  $28^{\circ}$  B. for sulphur oil and only at  $20^{\circ}$  B. for sedimentary and bleached oil.

Dissolve the 425 pounds of caustic soda in the kettle to a lye of  $25^{\circ}$  B. and allow the oil to run in a thick jet into the boiling lye. Saponification takes place at once, and when all the oil is in the kettle, a thick homogeneous soap is formed, which is allowed to boil a few hours. At this stage the soap shows a strong touch, which it loses, however, more and more, becoming harder in the same degree. When it is entirely homogeneous shut off the steam and add the common salt dissolved in water to  $23^{\circ}$  B. Now cover the kettle for one hour and then readmit the steam. The soap at once throws out clear lye, a thick grain free from froth being formed, which is ground, with constant boiling, by

sprinkling water over it until the soap slightly rises. These operations are carried on quite slowly, so that the soap up to this time has been boiling about eight to nine hours. The steam is then shut off and the thick and strongly-colored sub-lye allowed to settle and then drawn off or pumped out as much as possible. Direct as well as indirect steam is then again admitted and the soap again ground with water until it rises. In case it should paste, but a small quantity of salt water is required to restore it to its normal state. The sub-lye is examined, and, if strongly colored, again removed.

The soap should now show an improved color and pressure, have no touch of lye, and form an entirely clear grain. If such is not the case, it is again boiled with direct and indirect steam, only sufficient water and salt solution being added to reconvert it upon scant sub-lye into a clear curd-soap showing a firm pressure, which is again ground with water until the clear grain is liquefied and the lye separates from it in a thick, pasty state. Every soap-boiler must judge for himself of the condition of the soap in regard to neutrality, which, however, can be readily attained by frequent removal of the sub-lyes and grinding with water. Cover the kettle well and allow the soap to settle, and the longer this can be done the better it is.

*White olive-oil soap.*—The sedimentary oil from cold-pressed olive-oil is generally pure, the oil being repeatedly filtered before it is brought into the cisterns. Sedimentary oil from ordinary oils is, however, frequently colored and impure, and before use has to be heated and filtered, which in large factories is done by means of filter-presses. For white soaps, not only these sedimentary oils, but also bleached sulphur oils are used. These soaps are better or poorer in color and odor according to whether fine or ordinary oil is worked, and it may be asserted that the better in appearance the soap is the more claim it can lay to neutrality. The soaps from these oils are very sensitive towards lye and salt in the subsequent operations. They can be reduced to a considerable extent, forming nevertheless, when cold, a compact mass in which salt lye in an extremely finely-divided state is distributed.

Boiling is executed with caustic lye of 20° B., the oil being



allowed, as in the preceding soap, to run in a thick jet into the boiling lye. All the oil being in the kettle, a curly soap, frequently broken through like curd-soap, is formed. In this case it is allowed to boil quietly and continuously for several hours without an addition of salt being required. Water is then sprinkled over it, and if the soap contracts partially or entirely it is separated with salt solution of  $5^{\circ}$  to  $6^{\circ}$  B. until it boils high in the kettle. Grinding with water is then commenced in the same manner as with soap from sulphur-oil, except that the soap is always kept upon scant sub-lye, and weak salt solutions are added when required by the contracted soap.

The finished soap is covered warm, a coating of scum generally separating on the surface. Beneath this lies the bright white or yellowish soap, and between the latter and the lye a dirty soap.

The soap, after remaining in the kettle two to three days, is generally brought into small frames and crutched. The soap is usually sold as containing 62 per cent. of fatty acid, but as by the manner of preparation and grinding it mostly contains less in a fresh state, it is, after cutting, allowed to dry off before being packed. The fine appearance and smoothness which distinguish these soaps from all others are only acquired after storing in an airy cellar.

*Grain-soaps upon a precipitate of paste.*—First of all it is necessary to say that not too much palm-kernel oil or cocoanut-oil must be used in the preparation of these soaps. All fats, which by boiling upon sub-lye yield good soaps for the textile industries, can be used, about two-thirds of fats forming a ready union and one-third combining fat being generally taken.

The lyes for all these soaps being nearly the same, as well as the subsequent separation of the precipitate of paste, but few formulas are required to guide the soap-boiler in making similar combinations of available fats. The fats are saponified with caustic soda lye of  $25^{\circ}$  to  $28^{\circ}$  B. and the paste precipitated with salt solution of  $23^{\circ}$  B. We submit the following formulas:—

I. Tallow, cotton-seed oil, bone-fat, each 400 pounds, cocoanut-oil 500, caustic soda 375, salt 160.

II. Tallow 400 pounds, peanut-oil 600, bleached linseed-oil 200, palm-kernel oil 600, caustic soda 400, salt 180.

III. Bleached palm-oil 500 pounds, cotton-seed oil 400, bleached linseed-oil 300, cocoanut-oil 500, caustic soda 375, salt 150.

IV. Cotton-seed oil, peanut-oil, and bone-fat, each 400 pounds, palm-kernel oil 600, caustic soda 400, salt 175.

V. Saponified oleic acid 1000 pounds, tallow 400, palm-kernel oil 600, caustic soda 400, salt 200.

VI. Bone-fat 1200 pounds, cocoanut-oil 400, caustic soda 360, salt 150.

VII. Saponified oleic acid 1000 pounds, bleached palm-oil 200, cocoanut-oil 500, caustic soda 380, salt 170.

VIII. Saponified oleic acid 1200 pounds, cocoanut-oil 400, caustic soda 360, salt 150.

The boiling process is executed by first dissolving the required caustic soda in the kettle to a lye of  $25^{\circ}$  to  $28^{\circ}$  B. If much scrap is on hand, which is previously dissolved upon the lye,  $25^{\circ}$  B. is sufficient; with little scrap  $28^{\circ}$  B. Then introduce the fats and allow the mass to boil gently. When the last fat is in the kettle the proportions with a correct combination will approximately agree. It is, however, possible that there might be a trifle too much or too little lye which has to be adjusted by the operator. Boiling is now continued until a thick soap free from froth is formed, which is finally separated with salt solution until by the soap boiling thinner and fluttering when thrown with the spatula, it is observed that a precipitate of paste is formed when the addition of salt water is discontinued.

To obtain the soaps as neutral as possible moderate fitting and thorough evaporation are required, and if the subsequent separation with salt water be not carried to excess only the greater portion of the cocoanut-oil or palm-kernel oil soap, together with all dirt and coloring substances, will subside, while the separated soap is pure white and interspersed with silvery streaks. The finished soap is allowed to rest in the kettle a few hours, or, with large boilings, overnight, and is then brought hot into the frames and well covered.

Pure caustic soda lye and salt suffice, as a rule, for separation, and if the soap does not turn out good it is generally due to the base-soap having been too strongly fitted. It may, however,

happen that the combination is too weak, which is remedied by the addition of 20 to 25 pounds of crystallized soda, which is especially recommended when oleic acid or much bone-fat is used.

### *Barrel or Soft Soaps.*

*Natural grain-soaps.*—The result of a chemical examination of the best natural grain-soaps used in the textile industries would be fatty acids 45 to 46 per cent., potash  $12\frac{1}{2}$  to 13 per cent., water 41 to 42 per cent.

The raw materials required for the production of such soap are 48 per cent. of fat and oleic acid, 20 per cent. of 96 to 98 per cent. potash, 6 per cent. of lime for causticizing the potash, and water as much as required.

Such soaps are applied to many uses ; for washing yarns and cloth before and after dyeing and for fulling. Though they contain a large percentage of alkali, their solutions, as a rule, do not attack the colors more than those of hard soaps, which subsequently are frequently strengthened by the addition of crystallized soda. Wool treated with potash soap acquires a softer touch than with soda soap.

For the preparation of natural grain-soaps with faultless grain only lyes free from salts, hence at least of  $20^{\circ}$  to  $30^{\circ}$  B., can be used. If the weaker lye resulting from treating the potash with lime cannot be utilized for other soaps, the preparation of the lye must be either arranged accordingly or the weak lye evaporated to  $28^{\circ}$  B. Only pure high-grade potash can be used, 100 pounds of 96 to 98 per cent. potash requiring 33 to 35 pounds of well-burnt lime.

In the beginning of the operation the boiling of these soaps is executed by various methods, the final result being, however, always the same : the soap must be evaporated until it is sufficiently firm and by fitting induced to the regular formation of grain. The soap-boiler who carries on the initial boiling in a manner different from that here given and obtains good results may safely continue it, but to those less experienced the following formulas and method of boiling are recommended :—



I. Tallow 650 pounds, crude palm-oil 100, saponified oleic acid 750, cotton-seed oil 400, bleached linseed-oil 100.

II. Tallow, horse-fat, and saponified oleic acid, each 500 pounds, crude palm-oil 100, cotton-seed oil 400.

III. Tallow 400 pounds, bleached palm-oil 300, saponified oleic acid 700, peanut-oil 450, bleached linseed-oil 150.

IV. Tallow 300 pounds, pale bone-fat 700, crude palm-oil 100, saponified oleic acid 700, bleached linseed-oil or olive-oil 200.

Different formulas may be chosen according to the indicated proportions, it being, however, advisable always to use some crude palm-oil, as it gives to the soap a good appearance and odor. Good saponified oleic acid also imparts an agreeable odor to the soaps which is even perceptible in the solutions. Olive-oil, peanut-oil, sesame-oil, as well as cotton-seed oil, give abundant solutions, linseed-oil alone requiring precaution, as it is not much liked. Good qualities of bone-fat must be used, but not the product obtained by extraction.

Oleic acid forming a constituent of all the above formulas, the greater portion of the lye of 20° B. is brought into the kettle, a calculation being made of the quantity of lye required for the fats to be worked. With an average lye of about 24° to 25° B. (in this case one of 20° and 30° B.) 150 to 160 pounds will suffice for the saponification of 100 pounds of fat. If now, for instance, the formula calls for 700 pounds of saponified oleic acid, 1400 to 1500 pounds of lye of 20° B. are brought into the kettle, and after heating to boiling the oleic acid is gradually added. It saponifies at once. When all is in the kettle, boiling is continued quietly, and the other fats, first the fluid ones, such as linseed-oil, cotton-seed oil, and sesame-oil, and later on the bone-fat, horse-fat, and tallow are introduced together with the quantity of lye required for the fats. When the introduction of fats has progressed so far that the last fats, *except palm-oil*, have been reached, fitting must be already effected with the fats, *i. e.*, the touch must be so far reduced that the operator knows how much lye has to be added for the fats still wanting.

The soap is now boiled so thick that it "breaks into roses" and lies free from froth in the kettle, nothing further being ne-

cessary for this than that the soap is not too sharp, it being better to hold it somewhat weak, *i. e.*, so that the samples cool glassy. All other characteristics and advice found in old books, for instance, "the samples must be gray," "show a broad border of lye," etc., were well enough with the use of wood-ash, but are of no value at the present with the use of 96 to 98 per cent. potash. With good potash and good lime, lyes are obtained which give good soaps.

When the soap is evaporated, so that it forms a thick mass, and the soap-boiler thinks some lye be wanting, it is gradually added, however never more than 5 pounds at one time, moderate boiling being kept up, until a sample upon the glass remains warm for some time, and, when cooled, can be handled. Quiet ebullition is now continued until the soap is thickly evaporated, and rises with difficulty with an increased fire.

The time for the addition of the crude palm-oil has now arrived, for if this had been done previously, it would have become discolored and lost its pleasant odor. The crude palm-oil, which always forms a strong combination, is readily absorbed by the evaporated soap. The lye necessary for saponification is gradually poured into the quietly boiling soap, the kettle remaining half covered. 100 pounds of palm-oil require about 100 pounds of lye of 30° B. diluted with water to 20° B. When the soap is uniformly colored, samples are taken, and fitted with lye of 20° B., until there is a slight excess of alkali, which is indicated by a film on the surface of the cooled sample. A further indication of the soap being sufficiently fitted is when particles of froth appear on the sides, which do not disperse in spite of the gentle ebullition. With such lye as given above, the soap can be finished without the addition of carbonated lye. However, with a lye not as sure, the necessity of such an addition will be recognized after adding the crude palm-oil. If, after evaporation, the sample shows traces of sharpness, notwithstanding that the soap is hard, salts are wanting, and, as carbonates only are used for natural-grain soaps, potash solution is added.

Any defect caused by the use of too carbonated lyes also shows itself most fully towards the end of the operation. The soap may

boil thick and regular, and free from froth, but the samples, though showing a fine film on the surface, will lie flat upon the glass and congeal horny. This defect can only be remedied by pure caustic potash lye prepared either by dissolving caustic potash, or evaporating lye of  $10^{\circ}$  to  $12^{\circ}$  B. to  $30^{\circ}$  B. Such lye being, however, seldom on hand, it is best to keep back the crude palm-oil, evaporate the soap still further, and finally add lye of  $15^{\circ}$  to  $18^{\circ}$  B. (150 pounds for 100 pounds of palm-oil). The soap then contains a certain excess of alkali, of which the carbonates alone escape from the combination. If now the crude palm-oil be added, it recombines the soap, and improves it so far that it will pass muster.

After remaining for twenty-four hours in the uncovered kettle, the soap is poured into barrels and repeatedly crutched. After twenty-four hours the barrels are lightly covered, but headed only after most of the grain has formed.

*Olein soaps.*—In using oleic acid, it is first necessary to know whether it is the pure saponified article, though even in this case it is advisable to use other oil and some tallow in connection with it. Such formulas are :—

I. Saponified oleic acid 1500 pounds, tallow 400, crude palm-oil 100.

II. Saponified oleic acid 1550 pounds, crude palm-oil 100, cotton-seed oil 200, linseed-oil 150.

III. Saponified oleic acid 1450 pounds, peanut-oil 500, crude palm-oil 50.

IV. Saponified oleic acid 1500 pounds, crude palm-oil 100, olive-oil 200, sesame-oil 200.

As, by an addition of crude palm-oil, all these soaps acquire a uniform color, and their solutions become clear and full, the attention of the soap-boiler cannot be too often directed to the advantage derived from its use. And besides there is no other fat so well adapted for the purpose, and, even when used in small quantities, the soaps wash white, notwithstanding their yellow color.

The lyes used are the same as for natural grain soap, 40 pounds of potash being allowed for the saponification of 100 pounds of fat. Thoroughly evaporated soap gives, when fresh,



a yield of 238 to 240 per cent., though, in most cases, only 230 per cent. can be calculated on by the time the soaps are ready for the market.

The process of boiling is carried on in the following manner : 1000 pounds of lye are introduced into the kettle, and heated to boiling, and then 500 pounds of the oleic acid. A thoroughly saponified mass having been formed, 200 pounds more of lye are introduced, and then 100 to 200 pounds of oleic acid, until all is saponified. The oleic acid must be thus added in portions on account of the soap rising very readily. All the oleic acid having been saponified, the other fats are added, which remove the excess of lye, and the soap is then boiled thick and curly. Finally, the crude palm-oil is added, but only after the soap is free from froth and thoroughly fitted. The soap being thick, and breaking into roses, is then tested as to a correct fit. If a slight skin upon the surface of the cooled sample indicates sufficient fitting, and the soap is not soft to the touch, but glassy and firm, salts are wanting, and potash solution of 20° B. is added until it becomes softer. Frequently so much alkali is, however, introduced into the soap by these means as to require an addition of fat. It is, therefore, best to regulate the proportions before all the fats, and especially the crude palm-oil, have been added.

The finished soap is allowed to stand in the uncovered kettle and is repeatedly crutched through, and when cooled to from 133° to 145° F., poured into the barrels, repeatedly crutched, and when almost cool covered.

*Smooth oil-soap.*—For the preparation of this soap, which is much used for washing wool and also for fulling, there are many formulas, generally with linseed-oil as one of the ingredients. We submit the following :—

I. Distilled oleic acid and linseed-oil, each 800 pounds, cotton-seed oil 400.

II. Distilled oleic acid 700 pounds, linseed-oil 800, cotton-seed oil or peanut-oil 500.

III. Distilled oleic acid and sulphur olive-oil, each 600 pounds, linseed-oil 800.

IV. Distilled oleic acid and linseed-oil, each 600 pounds, cameline-oil or rape-seed oil 800.

Of pure oleic acid, which is also adapted for the purpose, a higher percentage may be used—for instance, 1700 pounds of oleic acid and 300 pounds of linseed-oil.

A small addition of crude palm-oil (about 25 to 30 pounds for the above formulas) is also recommended.

For the saponification of 100 pounds of oil 38 to 39 pounds of potash are required, which are set with 35 per cent. of lime. During the hot summer months 10 to 20 per cent. of crystallized soda may also be used for the subsequent reduction of the causticity.

For boiling, the greater portion of the lye of  $18^{\circ}$  to  $20^{\circ}$  B. is brought into the kettle, and after heating to boiling, the oleic acid and sulphur olive-oil are added. The touch is then regulated with linseed-oil and the soap evaporated until it “breaks into roses.” Samples of this soap showing a stronger fit, the causticity is so far regulated that the soap, though thick, is perfectly clear and soft to the touch. The rest of the oils and the lye proportionate to them are only introduced after regulating the soap or during that operation if much carbonate has been added.

By subjecting the oils and lyes to an accurate examination before use irregularities cannot occur, they being only possible with defective fats or turbid lyes. Before attempting large boilings a test should always be made on a small scale to see whether the fats and lyes are suitable for such soap. The finished soap is allowed to stand uncovered in the kettle, and when cooled to  $122^{\circ}$  F. is poured into barrels, which, if small, are covered and allowed to stand over night. Soap in large barrels is crutched through when cold, and after standing, tightly covered for a few days, the barrels are headed up.

## CHAPTER XVIII.

## MANUFACTURE OF TOILET-SOAPS, SHAVING-CREAMS, MEDICATED SOAPS, ETC.

FOUR distinct processes are in vogue for the manufacture of these soaps : 1, by the warm process ; 2, by so-called cold saponification ; 3, by remelting and subsequent perfuming and coloring of finished soaps ; and 4, by comminuting, perfuming, coloring, and subsequently pressing together by means of specially constructed machines, of so-called stock-soaps, carefully prepared for the purpose. The last are the most valuable and durable and at the same time the most expensive toilet-soaps manufactured. They require a great deal of manipulation by costly machinery, which has been chiefly devised by the French, although equally good, if not superior, machines are now constructed in this country and will be illustrated and described later on.

*Toilet-soaps by the warm process.*—By this process toilet-soaps are prepared either from cocoanut-oil alone or in connection with other fats and oils, such as tallow and castor-oil, by first melting the previously purified fats and then gradually saponifying them with caustic lye of  $20^{\circ}$  to  $40^{\circ}$  B. until completely saturated. As soon as the soaps have acquired the requisite consistency, which is eventually effected by the addition of solution of common salt and alkaline carbonates, they are colored if required, and after cooling to from  $144.5^{\circ}$  to  $167^{\circ}$  F., are brought into tight frames and allowed to cool as rapidly as possible without being covered. Many such soaps are brought into commerce, especially from Austria-Hungary, which are cheaper than all other toilet-soaps because the yield can be increased at will by the addition of solutions of common salt and potash. We give in the following a few directions for such soaps :—

*Cocoanut-oil soap with a yield of 300 to 320 per cent. by the warm process.*—100 pounds of cocoanut-oil are formed into a



paste with 30 pounds of potash-lye of  $15^{\circ}$  to  $16^{\circ}$  B., and after a slight combination is established, 60 pounds of soda lye of  $15^{\circ}$  B. and 140 pounds of  $20^{\circ}$  B. are added.

As the causticity of ordinary lyes varies very much, a test as to the touch must be made after the addition of the last lye and any defects corrected with cocoanut-oil or lye. The soap must, however, not show a very strong touch, but form a clear paste and draw threads. The whole is then allowed to boil quietly for some time to establish a complete combination, and such being the case, 16 to 20 pounds of pure white common salt are scattered over it for hardening and to obtain the requisite consistency. A sample taken after the complete solution of the salt, and allowed to cool in a small dish, should show a delicate white color when cut and be hard and tough, without, however, being crumbly or moist. When the soap shows these properties the kettle is covered in order to disperse the froth, and the soap allowed to cool to about  $167^{\circ}$  F. It is then carefully ladled from any sediment formed on the bottom and poured into the frames. About 14 ounces of essence of mirbane are required for perfuming the above quantity of soap. Such soaps normally fitted, *i. e.*, neither too fine nor too coarse, keep even in winter without sweating. Soaps of this kind filled to a still greater extent are frequently prepared in Austria-Hungary by saponifying each pound of cocoanut-oil with 2 pounds of slightly caustic lye of  $20^{\circ}$  B., then adding 1 pound of potash solution of  $20^{\circ}$  B., and filling with solution of common salt of  $15^{\circ}$  to  $20^{\circ}$  B. to a yield of 600 to 800 per cent. Such products, of course, do no longer deserve the name of toilet-soaps.

*Transparent soaps.*—The best toilet soaps prepared by the warm process are no doubt the *transparent soaps*, also called *glycerin soaps*. Formerly they were generally prepared with a large addition of alcohol, in consequence of which they dried out very strongly. Then came a time when the manufacturers went to the other extreme of using no alcohol whatever, but much alkaline carbonates, the result being a soap with a strong tendency to sweat. At present, very beautiful soaps are obtained from combinations in which a due medium is observed between the use

of alcohol and alkaline carbonates. We will illustrate this by the following formula:—

72 pounds of tallow and 64 pounds of cocoanut-oil, previously purified by remelting upon water, are first stirred together in the cold way with 106 pounds of soda-lye of  $38^{\circ}$  B. The soap is then left in the covered kettle to heat spontaneously, which, if necessary, is assisted by heating the kettle from the exterior by steam. If no steam can be had, it is best to prepare the soaps in a kettle suspended in a water-bath, since an open fire strongly affects their color. When the soap is combined so far as to lie in the kettle like a clear grain-soap, and has nearly reached the boiling point, a hot solution of sugar prepared from 60 pounds of sugar and 60 pounds of water is added to clarify the soap, 30 pounds of glycerin being introduced at the same time. Now stir the mass thoroughly, cover the kettle, and allow it to stand for two to three hours, the steam being, of course, shut off during this time. Then add to the soap, which will have become somewhat clarified during this time, for further clarification, 12 pounds of potash solution of  $16^{\circ}$  B., in which 10 pounds of crystallized soda have been previously dissolved. Cover the soap, allow it to stand for one hour, and finally add 20 pounds of alcohol, which will render the soap perfectly clear and short. After again resting for one hour, place a sample of the soap in the glass; it should be perfectly transparent and clear, and remain so after cooling, and show good pressure. The surface of the soap in the kettle should not be covered with a layer of undissolved grain, which would be due to too much lye or too little water, which, however, will be scarcely the case by following the directions; the soap will rather exhibit a light white froth resembling the foam of beer. The kettle is now allowed to remain at rest for some time to give any impurities a chance to subside. When the soap shows a temperature of from  $156^{\circ}$  to  $167^{\circ}$  F., it is poured into the frames. A suitable and, at the same time, not expensive perfume, which is added to the soap in pouring into the frames, consists of: Oil of citronella  $10\frac{1}{2}$  ounces, oil of fennel  $3\frac{1}{2}$  ounces, oil of verbena  $3\frac{1}{2}$  ounces, oil of peppermint 14 drachms, oil of lavender  $2\frac{1}{2}$  ounces.

Coloring is not necessary; if, however, desired, a solution of  $1\frac{3}{4}$  to  $2\frac{1}{2}$  drachms of uranin (aniline) orange in boiling water may be used.

The above formula for transparent soap may be modified in many ways by using more or less tallow, cocoanut-oil, or castor-oil, or by substituting sugar solution for a portion of the glycerin. We add a few formulas:—

*White alabaster soap.*—Stearin 13 pounds, cocoanut-oil 22 pounds, glycerin 13 pounds, lye of  $38^{\circ}$  B. 18 pounds, alcohol of 96 per cent. 26 pounds.

Saponify the stearin and cocoanut-oil, previously heated to  $178^{\circ}$  F., with the lye, and add the alcohol. After a combination is effected, which takes place almost instantaneously, add the glycerin. When the soap is clear, cover the kettle, and let it stand until cooled to  $133^{\circ}$  F., when it is brought into the frames, and perfumed with 4 ounces of oil of bergamot, 1 ounce of oil of geranium, 14 drachms of oil of neroli, and 1 ounce of oil of lemon. The soap is not colored, as it is to remain white, as indicated by its name.

*Eau de Cologne transparent glycerin-soap.*—Tallow 20 pounds, cocoanut-oil 12 pounds, castor-oil 8 pounds, lye of  $38^{\circ}$  B. 20 pounds, alcohol 20 pounds, glycerin 20 pounds, and 5 pounds of sugar dissolved in 5 pounds of water.

The process of preparation is the same as for alabaster-soap. The soap is perfumed with oil of bergamot  $8\frac{1}{2}$  ounces, oil of lemon 3 ounces, oil of lavender 11 drachms, oil of neroli 1 ounce, and 2 drachms of oil of rosemary and a few drops of oil of rose dissolved in 1 pound of alcohol of 69 per cent. It is colored with safflower.

*Transparent glycerin-soap.*—32 pounds of cocoanut-oil, 16 pounds of stearin, 3 pounds of tallow, and 1 pound of pale resin are brought into the kettle, heated to  $167^{\circ}$  F., and strained through a fine, white cloth. In the mean while, weigh off and heat also to  $167^{\circ}$  F. 28 pounds of lye of  $38^{\circ}$  B., dissolve 6 pounds of sugar in 9 pounds of distilled water, and heat 16 pounds of glycerin of  $28^{\circ}$  B. in a water-bath to  $167^{\circ}$  F.

When the fat has attained the above-mentioned temperature, add the lye in a fine stream with constant stirring, and then 30 pounds of alcohol of 90 per cent. When saponification is complete, add



the sugar solution and glycerin with constant stirring, which will render the soap perfectly clear. Now cover the kettle, and let the soap stand for about ten minutes, when a sample placed upon a glass-plate should, when cold, be perfectly clear and transparent. If, however, there should be the slightest turbidity, add gradually 1 to 2 pounds of distilled water of  $189.5^{\circ}$  F. with vigorous crutching.

*Toilet-soaps by the cold process.*—The manufacture of soaps by the cold process has already been given in Chapter XV., so that it will only be necessary to give some formulas.

*Bitter-almond soap* is prepared by saponifying 100 pounds of cocoanut-oil with 50 pounds of caustic soda lye of  $38^{\circ}$  B. and perfuming with 14 ounces of oil of bitter almonds. Essence of mirbane may be substituted for a portion of the oil of bitter almonds, the quality of the soap being however not as good.

*Filled bitter-almond soap.*—100 pounds of cocoanut-oil, 50 pounds of soda lye of  $38^{\circ}$  B., 20 pounds of water-glass compounded with 4 pounds of soda lye of  $38^{\circ}$  B., and 5 pounds of potash solution. Perfume : 14 ounces of essence of mirbane.

*Honey-soap.*—Cocoanut-oil 100 pounds, soda lye of  $38^{\circ}$  B. 50 pounds.

Perfume : Oil of citronella  $9\frac{1}{2}$  ounces, cassia-oil  $1\frac{3}{4}$  ounces, oil of peppermint 5 drachms, lemon-grass oil  $1\frac{1}{4}$  ounces.

Color :  $1\frac{3}{4}$  ounces of uranin (aniline) orange dissolved in boiling water.

*Honey-soap (filled).*—Cocoanut-oil 100 pounds, soda lye of  $38^{\circ}$  B. 50 pounds, tale 12 pounds, stirred into the warm oil before adding the lye.

Perfume : Oil of citronella 7 ounces, cassia-oil  $3\frac{1}{2}$  ounces, oil of cloves 1 ounce.

Color : 2 ounces of uranin (aniline) orange dissolved in boiling water.

*Bergamot-soap.*—Cocoanut-oil 16 pounds, lard 4 pounds, soda lye of  $40^{\circ}$  10 pounds.

Perfume : Bergamot-oil  $3\frac{1}{2}$  ounces, oil of geranium 11 drachms.

*Alpine-flower soap.*—Saponify 40 pounds of cocoanut-oil and 20 pounds of tallow with 30 pounds of lye of  $40^{\circ}$  B., and perfume the soap with 9 drachms of lemon-oil, 5 drachms of rose-

mary-oil,  $2\frac{1}{2}$  drachms of cinnamon-oil, 6 drachms of oil of peppermint, 5 drachms of oil of sage, and 6 drachms of lavender-oil, dissolving the oils in  $8\frac{1}{2}$  ounces of alcohol. Color the soap green with a composition prepared from two parts of indigo and one part of picric acid.

*Bismarck-soap*.—Cocoanut-oil 24 pounds, castor-oil 4 pounds, soda lye of  $40^{\circ}$  B. 14 pounds. Perfume with cinnamon-oil  $2\frac{1}{2}$  ounces, oil of cloves 5 drachms, oil of sassafras 8 drachms, oil of bergamot 5 drachms, oil of lemon  $1\frac{1}{2}$  ounces. Color dark with Bismarck brown.

*Brown eagle-soap*.—Stir together at  $90.5^{\circ}$  F. 40 pounds of cocoanut-oil with 23 pounds of lye of  $35^{\circ}$  B., color with 11 drachms of brilliant brown dissolved in  $1\frac{1}{2}$  pounds of boiling water, and perfume with 11 drachms of palma-rosa oil and 1 ounce 3 drachms each of essence of mirbane, lavender-oil, and cumin-oil.

*Pumice-soap*.—Ceylon cocoanut-oil 16 pounds, soda lye of  $40^{\circ}$  B. 8 pounds, pulverized pumice-stone 10 pounds. Perfume with oil of thyme  $1\frac{1}{2}$  ounces and oil of bergamot 9.5 drachms.

*Pumice-soap (another receipt)*.—Cocoanut-oil 30 pounds, soda lye of  $38^{\circ}$  B. 30 pounds, water-glass 30 pounds, pulverized pumice-stone 8 pounds, silver sand 34 pounds, indigo 1 ounce. Perfume with  $6\frac{1}{4}$  ounces of aniseed-oil.

*Bouquet-soap*.—Saponify 40 pounds of cocoanut-oil and 60 pounds of lard with 50 pounds of soda lye. Perfume with sassafras-oil  $3\frac{1}{2}$  ounces, bergamot-oil  $5\frac{1}{4}$  ounces, rose-oil  $2\frac{1}{2}$  drachms, oil of lemon  $3\frac{1}{2}$  ounces, oil of thyme  $7\frac{1}{4}$  ounces, oil of cloves 1 ounce, oil of neroli  $1\frac{1}{4}$  ounces, and tincture of musk  $1\frac{3}{4}$  ounces.

*Chinese-soap*.—Saponify 40 pounds of cocoanut-oil with 20 pounds of lye of  $38^{\circ}$  to  $40^{\circ}$  B., and perfume with oil of Portugal  $1\frac{1}{4}$  ounces, oil of bergamot  $1\frac{1}{2}$  ounces, oil of lemon  $1\frac{1}{4}$  ounces, tincture of musk 4 ounces, oil of patchouli  $2\frac{1}{2}$  drachms.

*Marsh-mallow (althea) soap*.—Saponify 20 pounds each of cocoanut-oil and tallow with 20 pounds of lye of  $40^{\circ}$  B. Perfume with oil of lavender  $5\frac{1}{4}$  ounces, oil of lemon 2 ounces, oil of bergamot 11 drachms, oil of peppermint 1 ounce, oil of neroli  $1\frac{1}{4}$  ounces, and color yellow with tompico yellow, or orange with tompico orange, or rose-red with brilliant rosa (aniline).

*Glycerin cold-cream soap.*—Melt 25 pounds of cocoanut-oil, 25 pounds of tallow, 8 pounds of castor-oil, and 2 pounds of crude palm-oil, heat to  $167^{\circ}$  F., and dissolve in the mass 15 pounds of scraps of white or yellow cocoanut-oil soap. Then stir in 30 pounds of lye of  $36^{\circ}$  B. until the soap-mass becomes thick, and add 1 pound of glycerin and 8 ounces of spermaceti, the last previously heated to  $167^{\circ}$  F. Now cover the soap, and after allowing it to rest for half an hour crutch thoroughly. After again resting for  $1\frac{1}{2}$  hours a thorough combination will be established, which is perfected by a steam or water-bath.

A clear yellow solid soap having the appearance of a smooth yellow wax-soap is obtained in this manner. After allowing it to cool somewhat it is brought into the frames and perfumed with cassia-oil  $1\frac{1}{2}$  ounces, cinnamon-oil 5 drachms, oil of cloves 2 ounces, oil of lavender  $3\frac{1}{2}$  ounces, tincture of benzoin  $3\frac{1}{2}$  ounces, oil of bergamot  $1\frac{1}{4}$  ounces, oil of winter-green 14 drachms, and tincture of musk  $3\frac{1}{2}$  ounces.

*Millefleurs-soap.*—Saponify  $12\frac{1}{2}$  pounds each of cocoanut-oil and olive-oil and 25 pounds of tallow with 25 pounds of caustic soda lye of  $40^{\circ}$  B., and perfume with bergamot-oil  $1\frac{1}{2}$  ounces, oils of cloves and of lavender each  $1\frac{1}{4}$  ounces, oils of cassia and of thyme each 11 drachms, and oil of neroli 5 drachms.

*Musk-soap.*—Saponify 40 pounds of cocoanut-oil with 20 pounds of caustic soda lye of  $40^{\circ}$  B. Perfume with  $3\frac{1}{2}$  ounces of tincture of musk, 2 ounces of oil of bergamot, and  $1\frac{1}{4}$  ounces of oil of lemon.

*Omnibus-soap.*—Cocoanut-oil 200 pounds, lye of  $20^{\circ}$  B. 275 pounds, common salt and potash each 15 pounds. Perfume with oil of mirbane.

*Rose-soap.*—Saponify 40 pounds of cocoanut-oil with 20 pounds of soda lye of  $40^{\circ}$  B., color with vermilion, and perfume with bergamot-oil  $2\frac{1}{4}$  ounces, geranium-oil  $2\frac{1}{2}$  ounces, and German rose-oil 10 drachms.

*Vanilla-soap.*—Cocoanut-oil 60 pounds, lard 30 pounds, crude palm-oil 10 pounds, pulverized cocoa 12 pounds, caustic soda lye of  $39^{\circ}$  B. 52 pounds. Perfume with vanilla  $1\frac{1}{2}$  ounces, balsam of Peru  $17\frac{1}{2}$  ounces, oil of lavender 3 ounces, and tincture of musk  $1\frac{1}{2}$  ounces.



*Vanilla-soap (superfine).*—Lard with vanilla 30 pounds, cocoa butter 10 pounds, palm-oil 10 pounds, caustic lye of  $36^{\circ}$  B. 26 pounds, wax 2 pounds, starch 2 pounds. Perfume with tincture of vanilla 4 ounces, tincture of musk 2 ounces, tincture of ambergris 2 ounces, and oil of rose  $\frac{1}{2}$  ounce.

Lard with vanilla is prepared by adding the vanilla to the lard (1 ounce to the pound), keeping it at a moderate heat for some days, straining, etc.

*Violet-soap.*—63 pounds of cocoanut-oil and 3 pounds of crude palm-oil are melted, cooled to  $108.5^{\circ}$  F., strained, and colored with  $2\frac{1}{2}$  ounces of vermilion. Then introduce into the fat through a hair-sieve, with constant stirring, 4 pounds of pulverized orris-root, 1 pound of pulverized orange-peel, and  $\frac{1}{2}$  pound of pulverized benzoin. When all the powder is dissolved, saponify the mass with 34 pounds of soda lye of  $38^{\circ}$  B. and perfume with lavender-oil  $2\frac{1}{4}$  ounces, bergamot-oil  $2\frac{1}{4}$  ounces, oils of cassia and of cloves, each  $1\frac{1}{2}$  ounces, and tincture of musk 2 ounces. The soap need not be colored, it naturally being of a beautiful brown color.

*Violet-soap (English).*—Tallow 30 pounds, cocoanut-oil 30 pounds, caustic soda lye of  $40^{\circ}$  B. 30 pounds, carbonated potash lye of  $30^{\circ}$  B. 1 pound, pulverized orris-root  $4\frac{3}{4}$  pounds, storax  $1\frac{1}{2}$  pounds, pulverized orange-peel 1 pound. Perfume: Sambul-root\*  $3\frac{1}{2}$  ounces, musk  $2\frac{1}{4}$  drachms, sugar color 1 ounce, oil of sassafras 8 drachms, oil of bergamot 3 ounces, oil of lavender 8 ounces, oil of orange 8 drachms.

The pulverized orris-root and orange-peel are sifted into the fat, and the storax, previously dissolved in some hot fat, is then added. The musk is triturated with some sugar, and the sugar color, previously mixed with  $4\frac{1}{2}$  ounces of water, added. The perfumed color is poured into the lye. The fat is then allowed to cool to  $88^{\circ}$  F. and the lye heated to  $68^{\circ}$  F.

The soap is not immediately brown, the color as well as the aromatic odor being brought out only after storing for some time.

*Windsor-soap (brown).*—Tallow, cocoanut-oil, and soda lye of  $37^{\circ}$  B., each 50 pounds, potash solution of  $15^{\circ}$  B. 4 pounds. Perfume with cassia-oil  $7\frac{3}{4}$  ounces, oils of cumin, cloves, and

\* The root of an umbelliferous plant, *Euryangium Sambul*.

lavender, each  $1\frac{1}{2}$  ounces, oil of thyme 1 ounce, and neroli petit grain  $1\frac{1}{4}$  ounces, and color with  $1\frac{1}{2}$  ounces of Hessel's leather-brown dissolved in boiling water.

*Windsor-soap (white).*—Saponify 20 pounds each of tallow and cocoanut-oil with 20 pounds of caustic soda lye of  $38^{\circ}$  B., and perfume with lavender-oil 4 ounces, oil of cumin  $4\frac{1}{4}$  ounces, citronella-oil 14 drachms, fennel-oil 11 drachms, and cassia-oil  $3\frac{1}{2}$  ounces.

*Toilet-soaps by remelting.*—In England the manufacture of toilet-soaps forms a special branch of industry. The manufacturer, or, to use a trade term, the remelter purchases the various soaps in their raw state from the soap-maker; these he mixes by remelting, and then perfumes and colors them according to the soap to be produced. The remelter does not confine himself to two or three varieties of soaps, but uses more than eight, mixing three or four for the different toilet-soaps.

These soaps are prepared from various fats and are known as

*Tallow-soap.*—This is a pure, solid, neutral soap prepared from fine tallow and pure caustic soda lye.

*Oil-soap*, as made in England, is a combination of olive-oil and caustic soda lye boiled to grain upon several waters and ground. It is very hard, contains but little water, and is of a greenish color.

*Castile-soap*, as imported from Spain, is also prepared from olive-oil and caustic soda lye, but is colored with sulphate of iron. The solution of this salt being added to the soap after it is manufactured, it is decomposed by the alkali present and ferrous oxide is diffused through the soap, giving it a black, marbled appearance. When the soap is cut up into bars the ferric oxide passes by absorption of oxygen into peroxide; hence a section of Castile soap shows the outer edge red-marbled, while the interior is black-marbled. Some Castile-soap is not artificially colored, but the marbling is produced by the use of barilla soda containing ferrous sulphide.

*Palm-soap.*—This soap was formerly boiled to grain, but is at present boiled to paste, crude palm-oil and soda lye of about  $24^{\circ}$  B. being generally used. Yield 220 to 225 per cent. It is generally used for filled toilet-soaps. The odoriferous principle of

palm-oil, resembling that from orris-root, remains intact in the presence of an alkali, the soap retaining the odor of the oil.

*Yellow-soap* is a cocoanut-oil soap with 20 per cent. of crude palm-oil and 10 per cent. of resin.

*Marine-soap*.—Though the preceding soap is of little value, this is still less so, as it is boiled from cocoanut-oil and soda lye, colored yellow with more or less palm-oil, and is made to yield 400 to 500 per cent. It contains besides a great excess of alkali and much water in combination.

*White fig-soap*.—Under this peculiar name is understood in England a soft soap prepared from olive-oil and potash, which has seldom a fine, clear, and transparent appearance. The saponification is in most cases defective and incorrect, the soap either containing free, uncombined oil or an excess of alkali. This is also the case with

*Naples soft soap*, which is prepared from fish-oil (mixed with Lucca-oil) and potash.

The above soaps constitute the base of all the perfumed toilet-soaps, which are mixed and remelted in the following manner:—

The bar-soap is first cut into thin slabs by a wire in the usual manner or converted into shavings. This comminution is absolutely necessary, because it would be difficult to melt a bar as a whole, soap being a bad conductor of heat. For remelting kettles of various sizes, holding from 30 to 3000 pounds, are used, and are heated either by a steam-jacket or a water-bath.

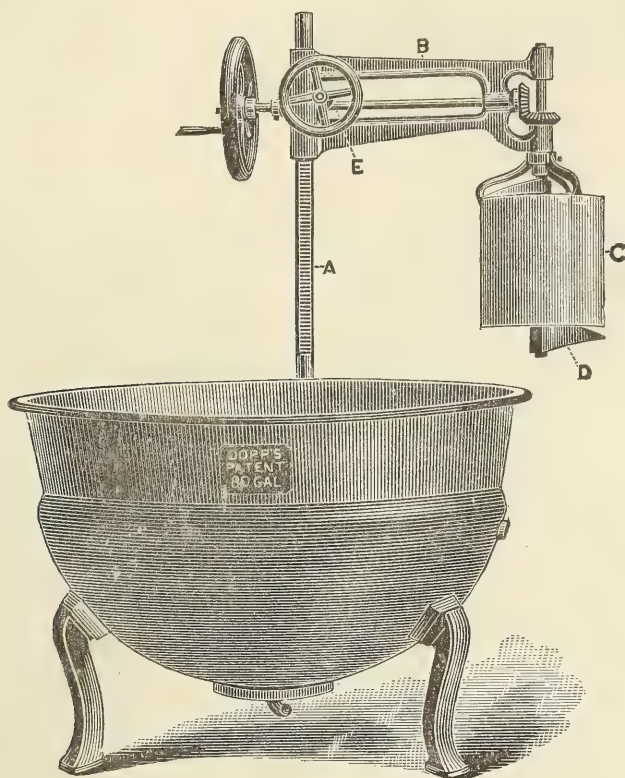
Fig. 76 shows an improved kettle, for rendering and refining toilet soap, which is manufactured by H. W. Dopp & Son, of Buffalo, N. Y. It is a seamless, steam-jacketed kettle, provided with an agitator so constructed that it can be easily removed from the kettle and swung out of the way when no agitator is required, or for cleaning the machine.

*A* is an upright provided with a rack screwed into a bracket cast on the kettle. A pinion, operated by a hand-wheel, *E*, engages with this rack, and thus the agitator can readily be raised out of the kettle. On reaching the top, it can be swung to one side out of the way, and the kettle can be used for boiling and all purposes to which a steam-jacket kettle can be put. The agitator is on the same principle as the remelting crutcher,



manufactured by the same firm, and described further on. It consists of a conveyor-screw, *D*, surrounded by a cylindrical casing, *C*. By loosening a set-screw, the conveyor-screw *D* can be withdrawn, and the whole machine can be cleaned very easily. It can be run by hand or power.

Fig. 76.



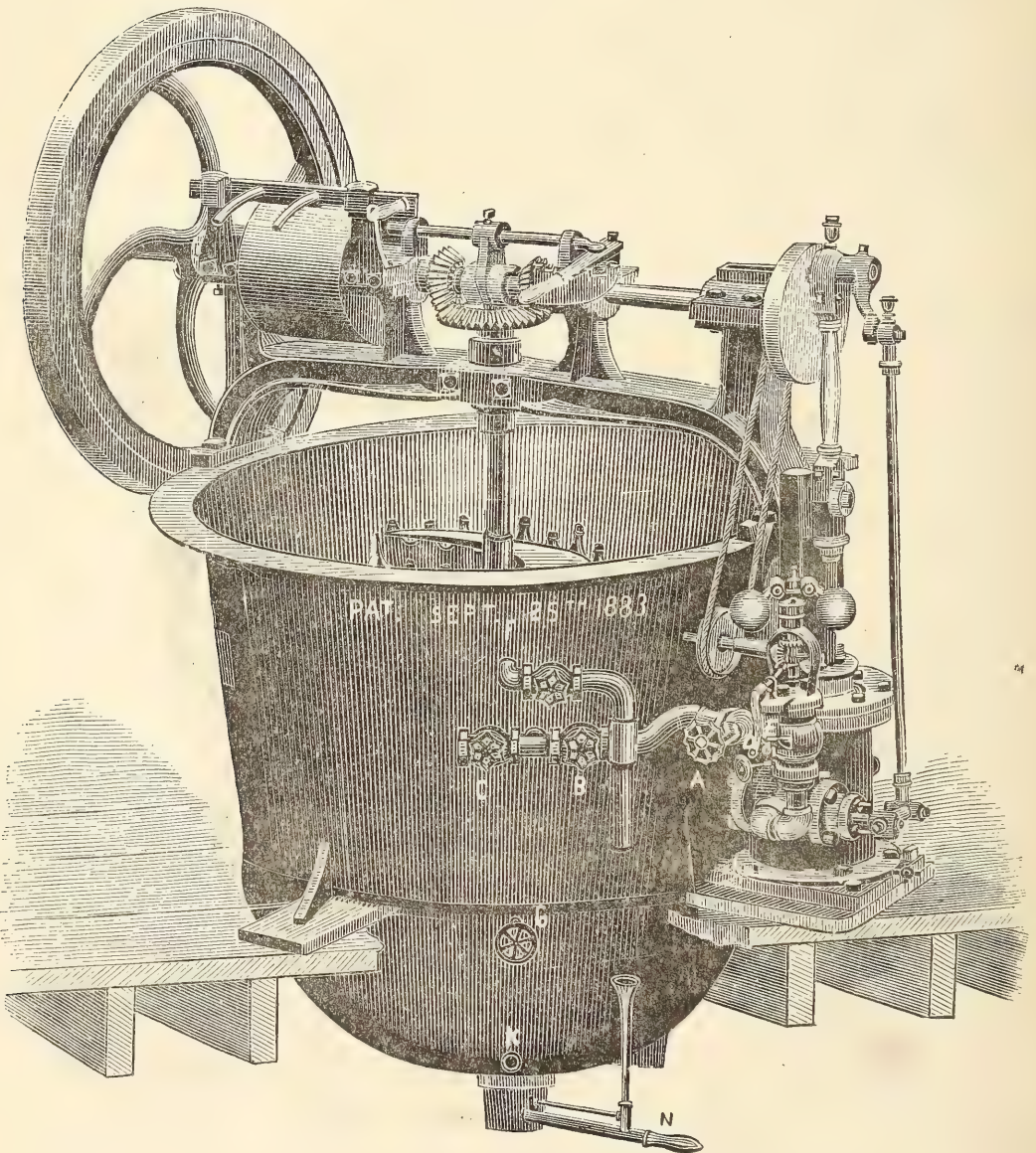
As the agitator can so readily be taken apart and thoroughly cleansed, and the machine has all the needed facilities for heating or cooling the mass by running cold water through the jacket, this kettle is very useful to soap-makers who make fancy toilet soaps.

Fig. 77 shows a remelting crutcher, with engine attached, manufactured by H. W. Dopp & Son, of Buffalo, N. Y., and Fig. 78 the same crutcher without engine.

The steam-jacket and inner shell are cast in one piece, having a number of stays between the inner and outer shell, the same as in the jacketed, seamless steam-kettle (Fig. 76); but the remelting kettle has a large outlet in the centre of the bottom for

the discharge of the contents. A steam-heating radiator, composed of a series of cylindrically arranged pipes having open spaces between them, is placed in the centre; through this radiator steam passes directly to the jacketed part of the kettle,

Fig. 77.

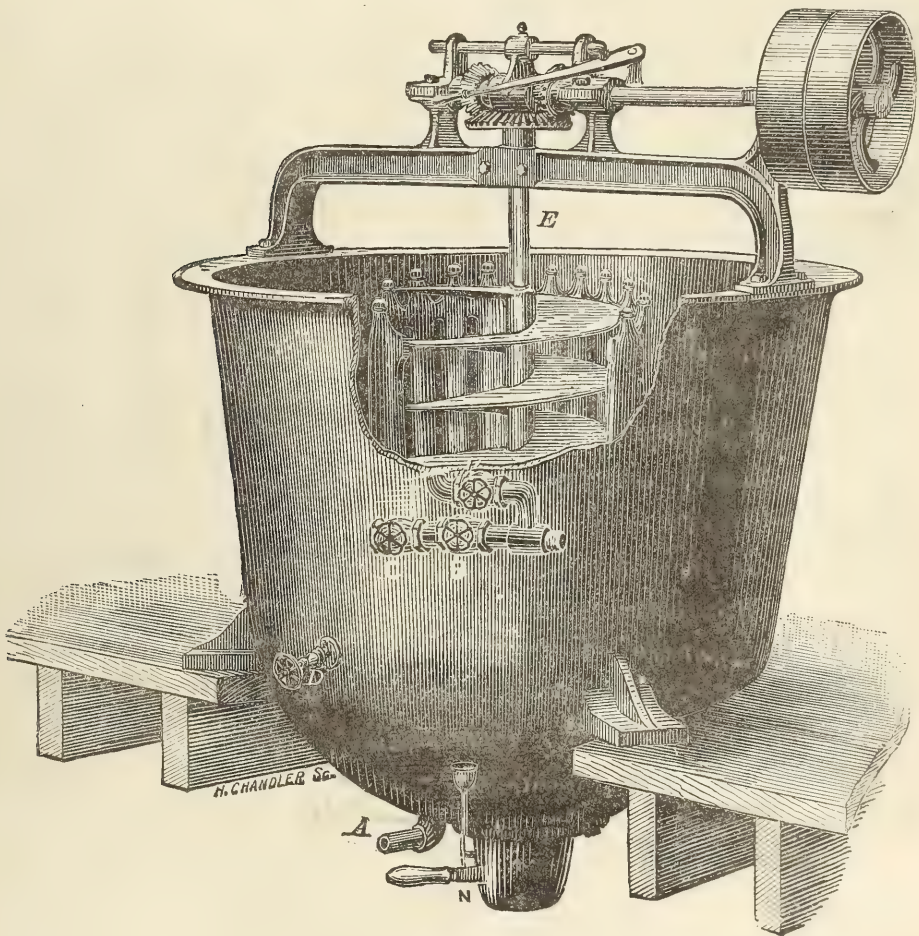


which can be cut off from steam supply, so that the inner cylinder only has steam. A conveyer-screw is placed in the centre of this radiator, which surrounds the screw. As soon as a portion of the soap is melted, the screw is set in motion, thereby lifting the



soap up, and dumping it over the top of the casing surrounding the screw, and the centrifugal force forces it out of, or through the open spaces left between the pipes. The large scraps are carried up, and are wedged in between the open ports, if we may so call them, at the upper end of the radiator (Fig. 77). The constant motion of the screw shears the pieces off; and thus, in a comparatively short time, the largest scraps are completely cut up, *and the whole kettle full of soap will be thoroughly melted and crutched ready for framing.* It should be observed that the transferring of the soap into a crutcher, after remelting the

Fig. 78.



same, is here overcome, and the *two operations are finished in one.* Owing to the open spaces left between the pipes composing the radiator, there is no splashing of the soap, soapine, olivine, etc., whatever, as in other machines, however fast the conveyer-screw



is worked. Moist steam may be passed at will through the soap-scrap, etc., to moisten them if necessary. Furthermore, if desired, cold water may be passed into the jacket and radiator to facilitate the cooling of the soap. The conveyor-screw is worked with a belt and pulley driven by power, and should make about 130 turns per minute. The conveyor-screw may be worked forward or backward by merely shifting the clutch that drives the bevel gearing (Fig. 78).

The machine is a soap remelter as well as a mixer and crutcher, and is especially adapted for making cold soaps. A fine grade of toilet soap, equal to milled soap, can be made with it in from five to ten hours, and at less than one-fourth the cost.

The kettle should be set up sufficiently elevated, so that a soap-frame can be conveniently placed underneath. This may be done either by placing the kettle on a platform of desired height, if the height of the room permits, or by suspending it through the floor above the one on which the soap is framed. When the latter way is chosen, the kettle may either rest on its four side-lugs upon the floor, or it may rest on its top rim or flange, so that it is but little raised above the floor. Be particular that the kettle is set level. It is advisable to use a 4-inch belt, and also to put a steam-gauge in front of the kettle, for which purpose will be found a  $\frac{1}{4}$ -inch gas-pipe hole in *T*, between valves *B* and *C*. Valve *B* admits steam to the inner cylinder, from which it may be admitted to the jacket by valve *D*. When valve *D* is closed, the jacket does not receive steam, but only the water of condensation from the inner cylinder. Valve *C* is for the escape of hot water when the machine is used for cooling. Valve *F* admits steam to the jet-coil used for passing moist steam through the dry scrap to replace water that has evaporated. Of the two one-inch holes in bottom, one is for the escape or waste pipe, the other for the introduction of cold water for cooling purposes. The bent  $\frac{3}{8}$ -inch pipe with air cock is to be screwed into the corresponding hole in the jacket. It has a branch for a steam-gauge if thought desirable. The small oil-tube and funnel are to be attached to the outlet valve, as shown in cut. If the cold water arrangement is not desired, one hole can be plugged up. Two semicircular wooden covers should be made to fit the top

of the kettle. Arrange the handle *N*, so that it comes to a stop when entirely open, and let it also come to a stop when it is entirely shut, lapping equally over the outlets. There will be found two  $\frac{5}{8}$ -inch tapped holes at the side of the bolts at the bottom of the kettle; also two  $\frac{5}{8}$ -inch studs; which are to be screwed in to fill the holes. This will set the valve at its proper place, when the handle *N* is placed in between.

In using the kettle, fill it well up with soap-scrap; turn on the steam, from 5 to 20 pounds steam pressure in the inner cylinder is best adapted for remelting; too high pressure may, perhaps, scorch the soap. Put on the wooden covers, and turn on the live steam from steam-jet coil by valve *F*. If desired, the jacket may be cut off from steam supply by closing valve *D*, or only as much steam as is desired may be used. For this purpose the steam-gauge, in communication with jacket, is to be attached. For some soaps this will be necessary, as they are apt to become spongy when lying too long in the hot jacket. The kettle should be covered during the time that live steam is introduced into the scraps, and so long as the crutcher is not running; thus the soap will become more thoroughly moistened, and no steam be thrown into the room. In about fifteen to twenty minutes, the soap will be well melted down: fill up again with more scraps. After a comparatively short time, the covers may be removed, and the crutcher set in motion for a few minutes, so as to mix the melted and the unmelted soap somewhat. Stop the crutcher, and replace the covers, and let the steam and the heat of the jacket, etc. do its work sufficiently, so that the scraps will be almost melted, before attempting to finish the work by crutching. When soap is *crutched too much or too long, and, also, when crutched while too thick, it will become frothy and airy*. To avoid this, soap must never be crutched longer than necessary; it is also necessary that the water lost from the soap since first made be added again to the same by the *application of moist steam*. In order to obtain the best results in remelting, different qualities of soap-scrap may require a different temperature or pressure in the inner cylinder and jacket of the remelter; the steam-gauge will always tell how that stands. (Little pressure, less heat; greater pressure, more heat, etc.) Any man with

common skill and good will will soon master the perfect use of the apparatus. It must *not be expected* that soap-scrap, after being remelted, will turn out precisely the same as the new soap (from which the scraps were taken) when first made. It must be remembered that soaps, or the scraps therefrom, are undergoing a constant chemical change, in the course of time, in their appearance, color, etc.—one quality of soap more, another less. Practical soap-makers who have been using remelters for years advise that, in order to obtain just the desired result in appearance, etc. of the remelted soaps, a little filling of some kind or another be added. When the soap-scrap in progress of remelting have reached that point that, if the unmelted scraps be worked and mixed with the melted liquid soap, a soap will be turned out as thick and no thicker than new made soap, then the moist steam may be turned off, and the crutcher set in motion. Crutch the soap as long as is required to effect a perfect mixing, *but no longer*.

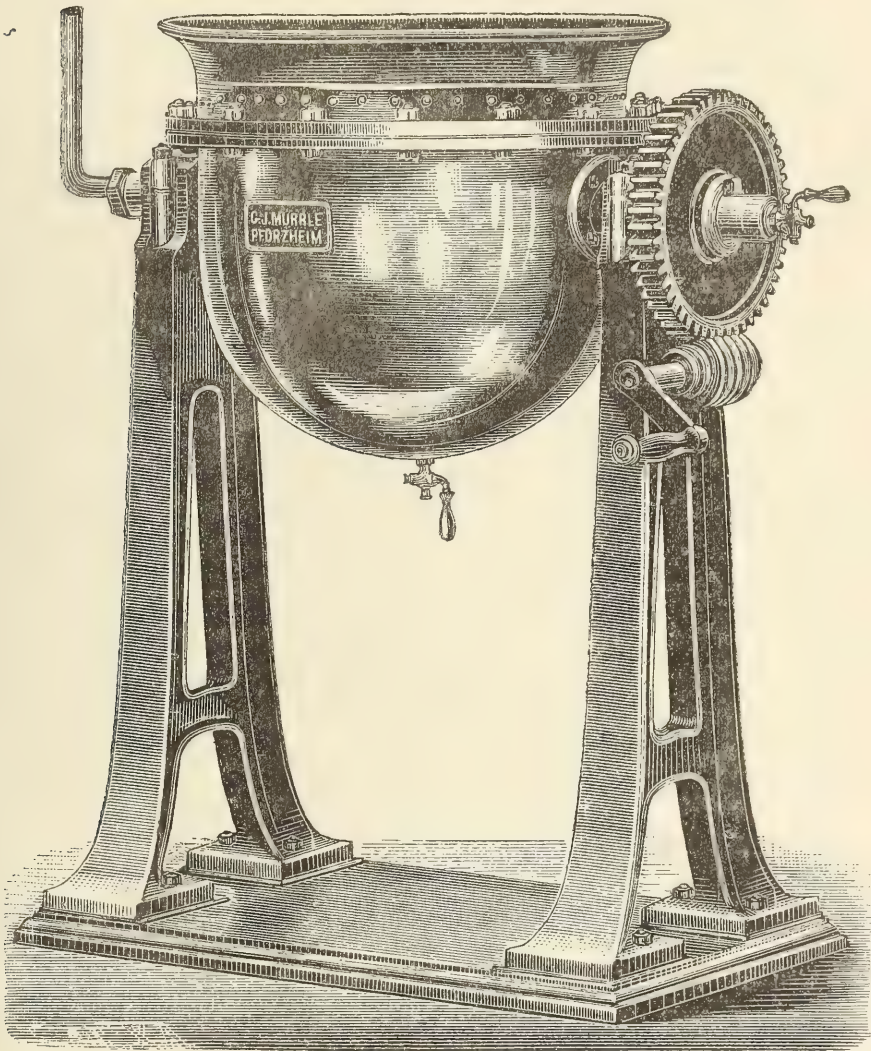
To make a toilet-soap equal to milled soap, introduce the soap-scrap and cuttings into the machine, turn on the steam, proceed as before, but *do not* use moist steam, as the object is to dry the soap, and run the conveyor-screw slowly. In five to ten hours can thus be produced a soap in every respect equal to a milled soap at less than one-fourth the cost. The bevel gearing of the driving shaft is to be *reversed*, by shifting the clutch of the gearing, before the finished soap is let out through the bottom valve. The reversing makes the screw run in the contrary way, and thus will force the soap down through the bottom of the kettle. Before reversing the gearing always stop the crutcher, otherwise you will be sure to break the gearing. When discharging the soap run the conveyor-screw backwards and at a slow speed; in this way the soap will be turned out quicker. To run at slow speed shift the belt only partly on to the fast pulley. In this way almost any speed from full speed to nothing can be secured. Should the soap be found too hot, turn on the cold water while the crutcher is working. When cold water is turned on the steam supply must first be turned off.

Be especially careful not to neglect to oil the bevel gearing and the journals thereof; if running dry, they will soon wear, and it



will require more power to run the crutcher. When steam is turned on be sure and have the condensed-water discharge-valve at the bottom of the steam-jacket partially open, so that a little steam will ooze out with the discharge-water. When the apparatus is not used, have the condensed-water discharge-valve *entirely* open to avoid water collecting in the jacket in case the steam-valve be leaky. Should water collect, it might freeze in

Fig. 79.



Steam-Jacket Remelter.

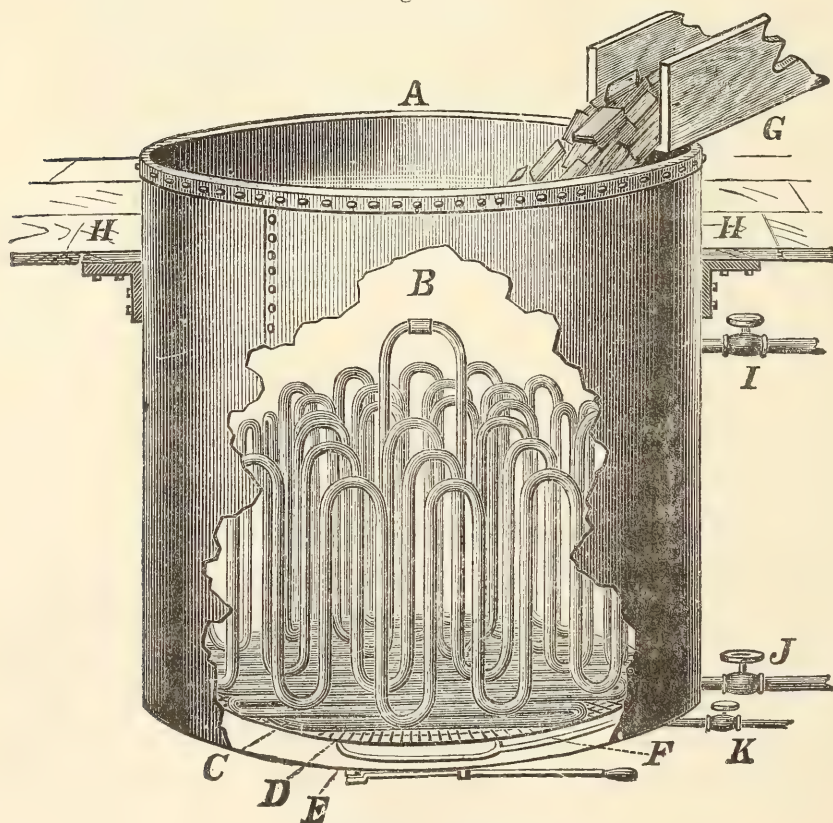
winter and thus destroy the jacket and the kettle. On first turning on steam open valve *D* and air-cock of jacket to get rid of

air. After all the air is out valve *D* must be closed, if no steam is wanted in jacket. After using the machine and before it is cold, blow out jet-coil by opening valve *F*; otherwise soap may solidify in the coil and give trouble on next using the machine.

A convenient form of a steam-jacketed kettle for remelting soap is shown in Fig. 79. It is provided with a tilting arrangement and rests by means of two pivots upon a cast-iron frame secured by screws to a heavy cast-iron base-plate. The steam is admitted by means of a stuffing-box through one of the pivots, and by turning the crank even the heaviest kettle can be readily tilted and emptied without unscrewing the steam-pipe or interrupting the admission of steam.

Whitaker's patent remelter with continuous coils of steam-pipe is shown in Fig. 80. It can be used for remelting soaps to be

Fig. 80.



Whitaker's Remelter.

converted into toilet-soaps and for remelting scraps, especially of soaps filled with water-glass, sal soda, and other substances. In



remelting such scraps by the boiling process all the filling would be lost, which with the immense quantity of filled soap manufactured at the present time would prove quite a loss. The following is a description of the above machine: *A*, wrought-iron cylinder with dishing-bottom; *B*, coil of continuous steam-pipe; *C*, horizontal scroll-pipe connecting with the upright pipe; *D*, wire diaphragm which serves to separate the soap; *E*, gate for discharging soap; *F*, small pipe for admitting direct steam through perforations into the body of the soap; *K*, inlet-pipe and valve for direct steam; *I*, inlet-pipe; and *J*, discharge-valve for condensed steam; *HH*, floor of building; *G*, spout for running in the scrap.

For use fill the remelter with the soap, put on the cover, close the bottom slide, and let on the open steam until the soap begins to melt, which will depend upon the dryness of the soap. When sufficiently melted shut off the open steam; open the bottom to drain off the condensed steam. Then let on the steam through the coils and put the frames in place to catch the melted soap. When in the frames stir well when half full, and also when full, in order to insure a uniform soap.

This machine will hold about 1000 pounds of soap and can be used six or eight times a day.

In remelting stock-soaps the shavings are put into the kettle by degrees, or what is technically called "rounds," *i. e.*, the shavings are placed perpendicularly all round the side of the kettle, a little water being added at the same time, the steam of which assists the melting. The kettle being covered up, in about half an hour the soap will have melted. Another round is then introduced and so continued until the entire lot of soap is melted. The more water the soap contains the easier it is melted; hence marine-soap or yellow-soap will melt in about half the time required for curd-soap.

When different soaps are being remelted to form one kind of toilet-soap, the various soaps are to be put into the kettle in alternate rounds. As the soap melts it is mixed and lumps are broken up by crutching. When the soap is all melted it is colored, if required, the perfume added, and after mixing the whole thoroughly by crutching, it is brought into the frames, the sec-



tions of which are frequently made of the width of the intended bar of soap.

*Almond-soap*.—White tallow curd-soap 100 pounds, oil-soap and cocoanut-oil soap, each 14 pounds, oil of bitter almonds 26 ounces, oils of cloves and caraway, each 8 ounces, or instead of the latter, rose-oil  $\frac{1}{2}$  ounce.

It is best, first to melt one-half of the tallow-soap, then the other two soaps, and when they are melted add the other half of the tallow-soap, and finally the perfume. By substituting nitrobenzole (essence of mirbane) for all or a portion of the oil of bitter almonds, a cheaper soap of not as fine a quality is obtained.

*Camphor-soap*.—Tallow curd-soap 100 pounds, oil of rosemary  $4\frac{1}{2}$  pounds, camphor  $4\frac{1}{2}$  pounds.

Reduce the camphor to powder by triturating it with some almond-oil, then sift it. When the soap is ready to be brought into the frame add the camphor and rosemary-oil, with vigorous crutching.

*Honey-soap*.—Best yellow-soap 50 pounds, fig-soap 7 pounds, oil of citronella  $1\frac{1}{2}$  pounds, saffron 1 ounce.

*Pumice-soap*.—White tallow curd-soap 10 pounds, cocoanut-oil soap 3 pounds, finely-pulverized pumice-stone 14 pounds, French lavender-oil 2 ounces, oil of geranium 1 ounce.

*Sand-soap*.—White tallow curd-soap and cocoanut-oil soap, each 7 pounds, fine quartz sand 28 pounds, oils of thyme, cassia, caraway, and French lavender, each 1 ounce.

*Windsor-soap (old brown)*.—This popular soap is made in the following manner: Convert 50 pounds each of palm-soap and half palm-soap into shavings and spread upon sheets of strong paper to dry; when dry melt in a water-bath with a small portion of an aromatic water, and when it is hard enough cut into shavings as before, drying it again and remelting and adding sugar color. After the third operation add the following perfume for the 100 pounds: Oil of bergamot 4 ounces, oils of caraway and cassia, each 2 ounces, lavender 8 ounces, cloves and petit grain, each 1 ounce.

Brown Windsor-soap owes its fine emollient properties to the amount of labor employed in its manufacture, for it is needless to

say that the more soap is worked and handled and melted and remelted the better it becomes.

*Windsor-soap (brown).*—White tallow curd-soap 75 pounds, cocoanut-oil soap, yellow-soap, and oil-soap, each 25 pounds. Perfume with oils of caraway, thyme, cassia, petit grain, cloves, and French lavender, each 2 ounces, and color with  $\frac{1}{2}$  pint sugar color.

*Windsor-soap (white).*—White tallow curd-soap 100 pounds, cocoanut-oil soap 21 pounds, oil-soap 14 pounds. Perfume with oils of caraway, thyme, and rosemary, each 26 ounces, oils of cassia and cloves, each  $4\frac{1}{4}$  ounces.

*French system of making toilet-soaps or milled-soaps.*—These are the best and finest toilet-soaps, hence it is of great importance that the so-called stock-soaps used in their preparation should be made from fresh and pure materials and contain no incompletely saponified fats, which would exert an injurious effect upon their durability. They must, therefore, be carefully saponified, salted out, boiled clear, and ground, which will be briefly described further on. Stock-soaps, if allowed to remain too long in blocks after being taken from the frames, readily turn rancid; hence they should be cut into bars as soon as possible, and these dried in the air or the drying-room.

The first operation is to “strip” the stock-soap, *i. e.*, to cut it up into strips or shavings. This may be done either by hand, which is rather a primitive way, though suitable and economical for small lots of soap, or by a machine called a chipper or stripper.

The tools required for working by hand consist of an ordinary carpenter’s plane and a good marble mortar and pock-wood pestle. Each end of the plane should be provided with a contrivance, so that when placed over the mortar it remains firm and is not easily moved by the parallel pressure of the soap against the projecting blade.

The operation commences by weighing off determined quantities of the soap that is to be cut up and perfumed. The plane is then laid upside down across the mortar and the bar of soap pushed across the plane until it is reduced to fine shavings. Soap as generally received from the soap-maker is in proper con-

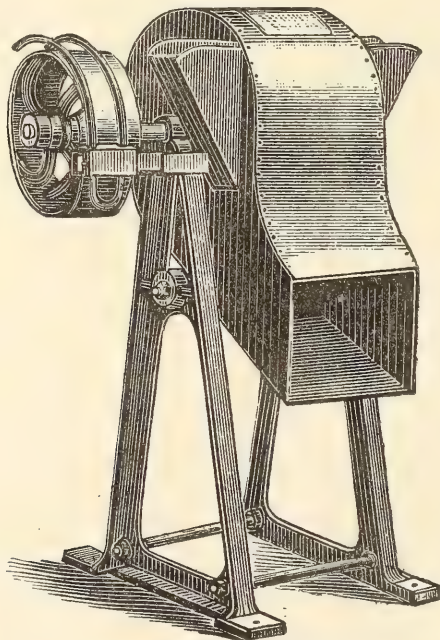
dition for thus working ; but if it has been in stock any time it is apt to become too hard, and after having been converted into shavings must be sprinkled with some distilled water and allowed to stand 15 to 24 hours to give the shavings time to absorb the water before the perfume is added.

After determining the size the cakes of soap are to be, what they are to sell for, and what they are to cost, the maker can measure out his perfume.

The soap being in proper condition in regard to moisture, etc., is now to have the perfume well stirred into it. This is done by working it thoroughly with the pestle for a few hours, when the soap is generally expected to be free from streaks and of uniform consistency.

The soap thus perfumed is then weighed out in quantities as required for the tablets and moulded by the hand into egg-shaped masses, which are laid separately in rows on a sheet of white paper, and allowed to dry for a day or two to fit them for the press. It is usual before placing the cakes in the press to dust

Fig. 81.



Soap-Stripper or Clipper.

them over with a little starch-powder, or very slightly to oil the mould, to prevent the soap from adhering to the letters or embossed work of the mould.



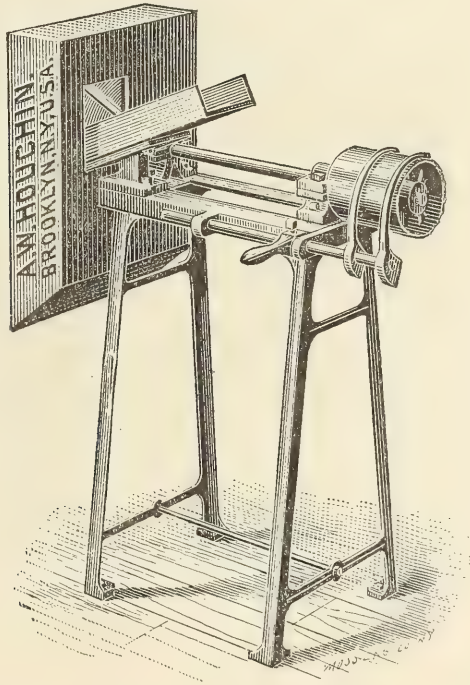
For the preparation of large quantities of toilet-soap it is more convenient and economical to use machinery.

The stock-soap is cut into shavings by a machine called a "stripper" and driven either by hand or power.

There are four or five different kinds of this machine, though the essential parts of all of them are one or two revolving disks provided with four to six knives and a hopper to contain the bars. Fig. 81 shows such a machine with two disks. Opposite the disks is a hopper or cylinder in which the bars of stock-soap to be stripped are placed. By pressing the bars against the disks, they are cut into shavings which fall through slits in the disks into a receptacle.

Fig. 82 is a power-chipper manufactured by Alfred Houchin, of Brooklyn, N. Y. The soap is cut into bars six inches square and, fed into the hopper and the disk, which is a revolving plane, converts it into shavings. The machine can also be arranged for

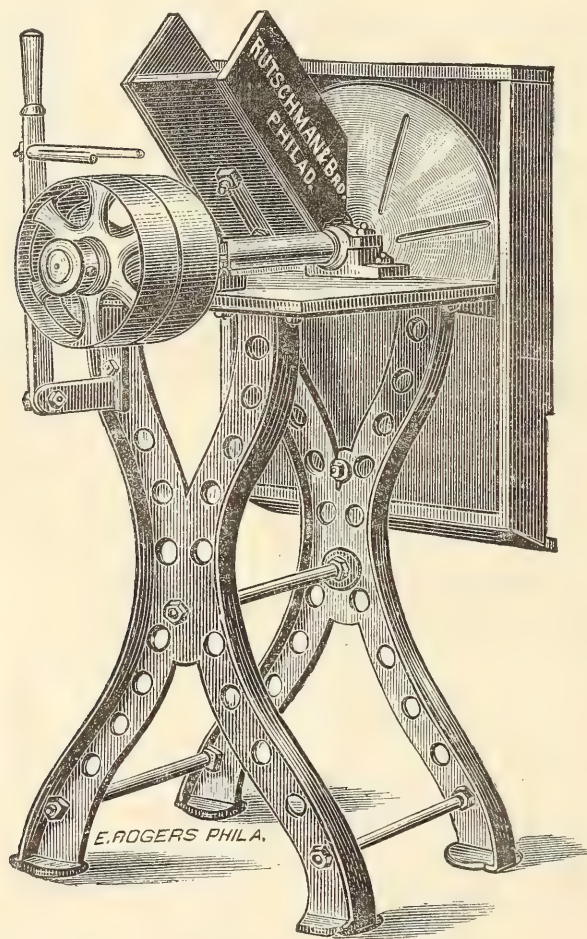
Fig. 82.



foot-power by taking off the pulleys and placing a fly-wheel in their place, which is connected to a treadle working the same as a foot-lathe or grindstone.

The latest and most improved machine for this purpose is the automatic soap-chipper manufactured by Messrs. Rutschman Bros., of Philadelphia. This chipper or stripper, Fig. 83, works with great rapidity, and is also adapted for laundry and other purposes. The plate has six knives, which can be regulated to cut the shavings of any thinness and can be cleaned with ease.

Fig. 83.



Rutschman's Automatic Soap-Chipper.

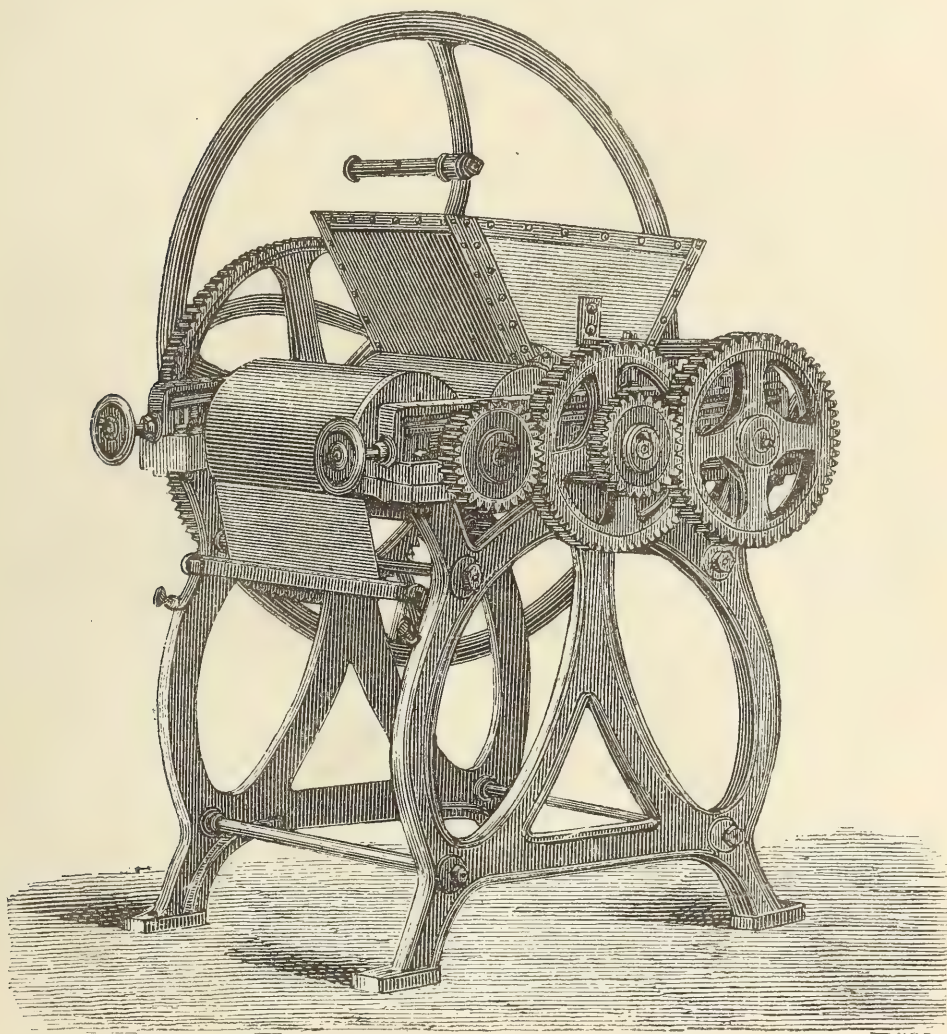
In working it is only necessary to lay the bars in the trough leading to the knives, as the machine feeds the soap against the knives automatically.

After stripping the soap, the shavings are frequently dried somewhat and are then brought into a wooden box lined with zinc or lead. The proper proportions of volatile oils and coloring matter (except when the soap is required to be white) are

first mixed in a separate vessel with a little alcohol, and the mixture is then added gradually to the shavings with constant stirring. The shavings are then conveyed to the

*Soap-mill* to be blended into a thick homogeneous paste. The mill is similar in construction to a cocoa-mill, and consists essentially of three or four contiguous rollers by whose action the

Fig. 84.



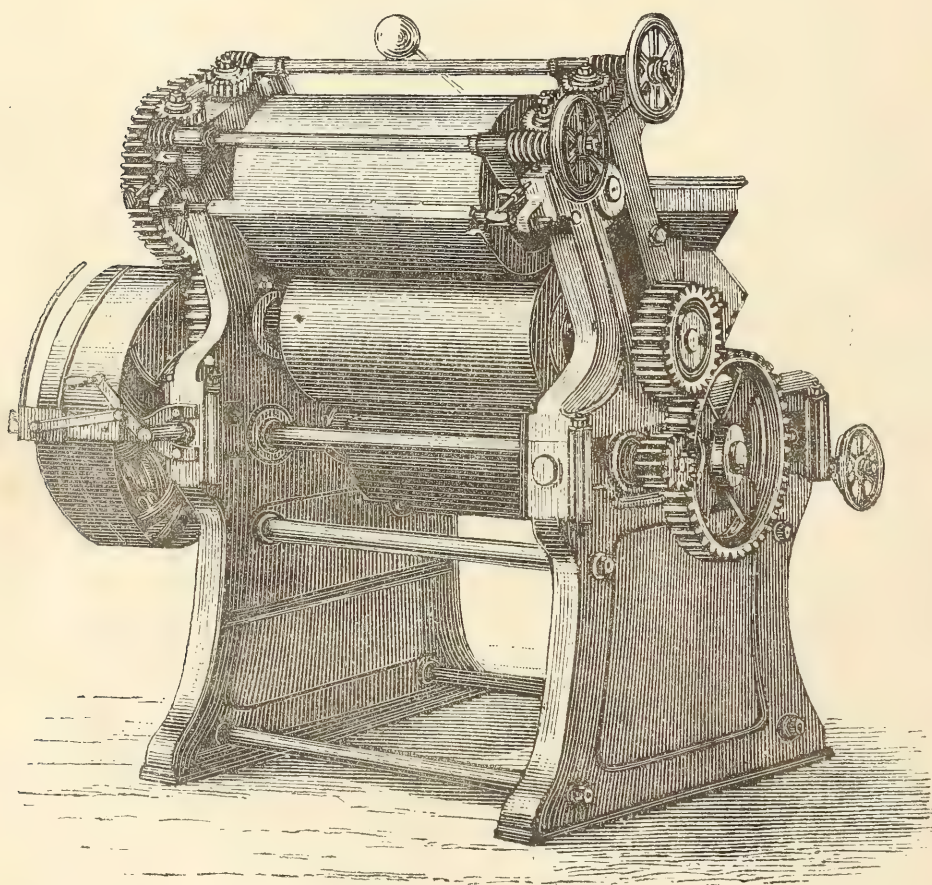
shavings, color, and perfume are intimately united. The size of the mill, and, whether it is to be worked by hand or steam, depends on the size of the establishment. Fig. 84 shows a mill with three rolls to be worked by hand. The rolls are of best syenitic granite finely polished. The perfumed and colored



shavings are brought into the hopper, and after passing through between the first two rolls fall upon the third. A scraper pressing against the third roll scrapes off the soap which falls into a receptacle. The milled mass must be returned to the hopper and again passed through the rolls until it is perfectly homogeneous.

With a machine with four rolls, Fig. 85, this repeated passing of the milled mass through the rolls is not required, since the operation is effected by the fourth roll. Besides, in large factories

Fig. 85.

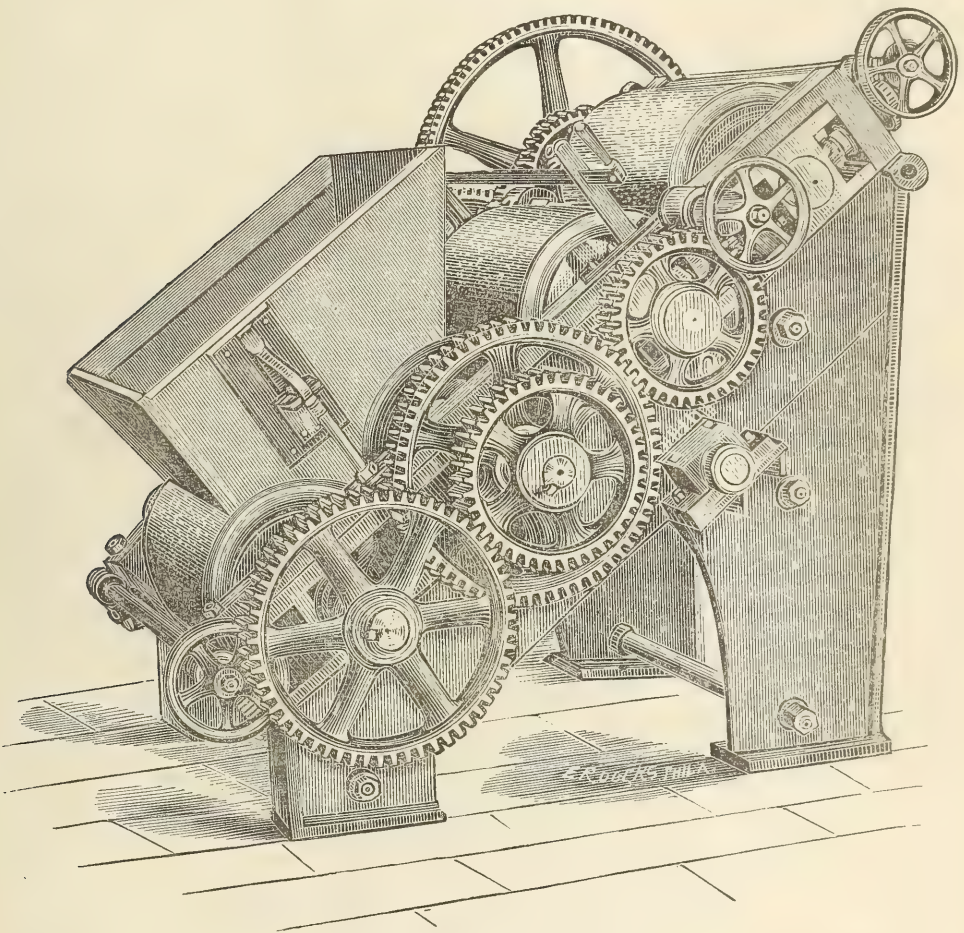


where the work is carried on continuously, two or three machines may be placed alongside each other and the thorough mixing of the mass effected by passing it successively through the machines.

The soap-mill manufactured by Rutschman Bros., of Philadelphia, and shown in Fig. 86, is constructed upon entirely new patterns and principles. The bed is of box-section extending to

the ground and on an incline, and with long bearings for the stone rollers, which are made from the best Quincy granite, a material well adapted for the purpose on account of its hardness; the surface finishing true and smooth, yet porous, so as to grind the soap quickly and perfectly. The shafts, which extend entirely through the rollers, are of steel and very heavy, so as to insure against all possibility of springing, and are fastened in the stone rollers with a special device which renders it impossible for them to become loose on the stone. The gearing and all the

Fig. 86.



Rutschman's Soap-Mill.

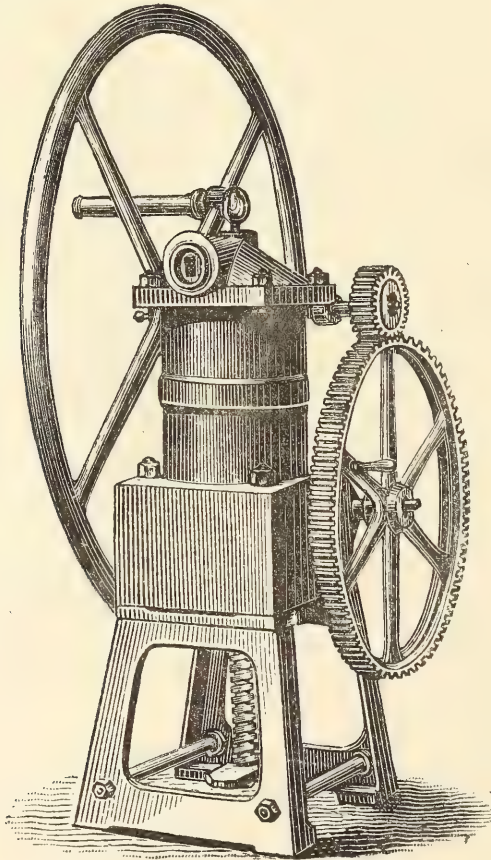
wheels are either machine moulded or machine cut. The pulleys are of large diameter and breadth and capable of transmitting ample power to the working parts of the machine. The stone rollers are automatically adjusted by a wheel and worm, which



motion is positive, insuring the whole length of the rollers working accurately together, by which the soap is ground thoroughly and rapidly and with the least possible wear on the machine. The mill has a capacity of 250 pounds per hour ; power required, 12 horse-power.

When a homogeneous mass free from grains and streaks has been formed, the soap is ready for the operation known as “plotting” or “peloting,” in which the paste is subjected to enormous pressure, sometimes 3000 to 4000 pounds to the square inch, to form it into continuous bars of any shape desired from which cakes may be cut. Various machines called “plodders” are

Fig. 87.



Soap-Plodder.

used for this purpose. Fig. 87 shows a plodder worked by hand, in which the pressure upon the soap in the vertical cylinder is effected by means of a piston. The milled soap is rammed into



the cylinder by means of a pestle, and pressure brought to bear upon the contents. The bar (of any shape desired, round, oval, or angular), coming from the discharge aperture, passes on to a table placed in front of the aperture, where it is cut into cakes.

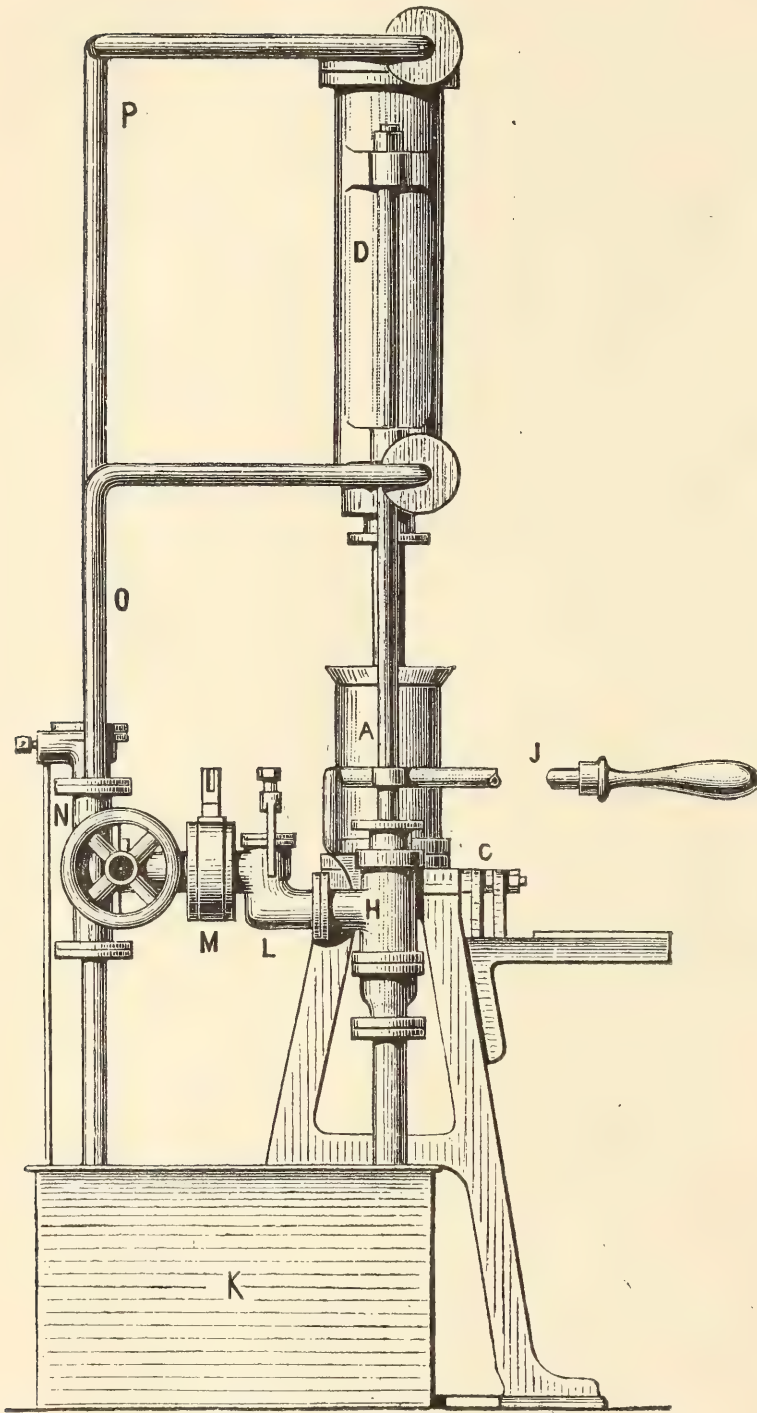
Another machine of the same order is provided with a mechanical ramming and cutting apparatus. The milled soap is simply poured into a hopper over the cylinder. A screw in the cylinder effects the pressure of the soap, which, passing through a sieve into the discharge aperture, comes out in bars of any desired shape. These machines can be provided with a heating apparatus (water, gas, or steam) so as to polish the surface of the bar leaving the machine.

Figs. 88 and 89 show a hydraulic plodder. A cast-iron cylinder serves for the reception of the soap-shavings. The lower end of this cylinder ends in a rectangular channel, *B*, to which a mould (of any shape the bar of soap is to have) is secured. The aperture of the mould is first closed by a plate, *C*, to allow of the soap-shavings brought into the cylinder being compressed to a compact mass.

With the cylinder *A* is connected by means of columns the water-cylinder *D*, in which moves the ram *E*. The latter and the press-ram *F* have a joint piston-rod, *G*. *H* is a pump which, when set in motion by means of the hand-lever *J*, sucks water from the reservoir *K* and conveys it to the cylinder *D*. A safety-valve, *M*, inserted behind the delivery-valve *L* prevents the water-pressure from exceeding a determined limit. As the rams have to be forced up or down by the pressure of the water, the latter is distributed in the box *N* by means of a slide into the pipes *O* and *P*, which conduct it to the water-cylinder *D*. Thus, with the aperture of the mould closed by the lid *C*, the soap-shavings are compressed into a compact mass and, after the removal of *C*, forced out as bars.

The so-called Boudineuse plodder, constructed by W. Rivoir, is intended for very large establishments. The machine is shown closed ready for work in Fig. 90 and open in Fig. 91. The principal part of this machine is the Archimedean screw-shaft, which possesses a progressive upward motion and is surrounded with a parabolical jacket. By this and an arrangement called

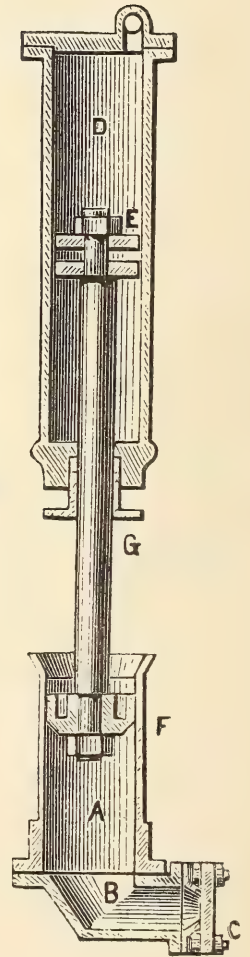
Fig. 88.



View.

Hydraulic Plodder.

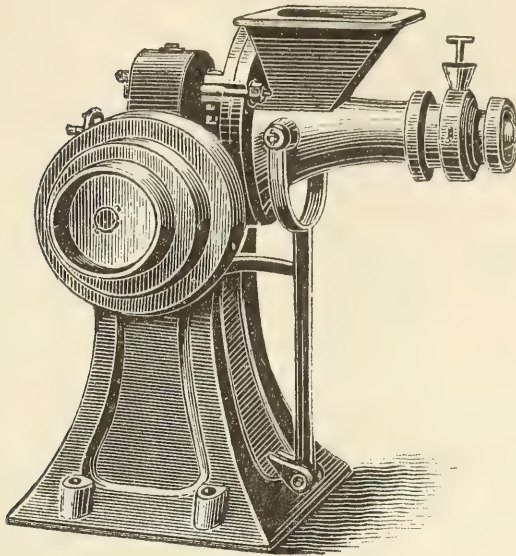
Fig. 89.



Section through Cylinder.

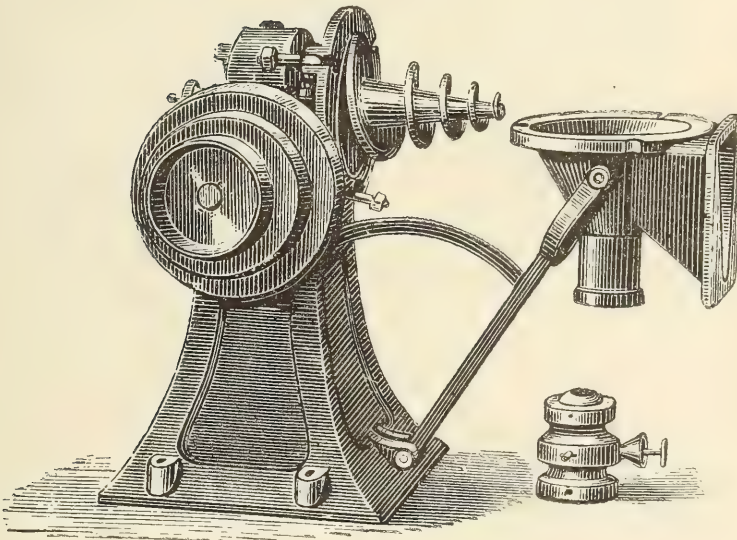
*A*, cylinder; *B*, mould; *C*, lid; *D*, water-cylinder; *E*, ram; *F*, press-ram; *G*, piston-rod; *H*, pump; *J*, hand-lever; *K*, reservoir; *L*, delivery-valve; *M*, safety-valve; *N*, distributing-box; *O P*, pipes.

Fig. 90.



Boudineuse Plodder, closed.

Fig. 91.



Boudineuse Plodder, open.

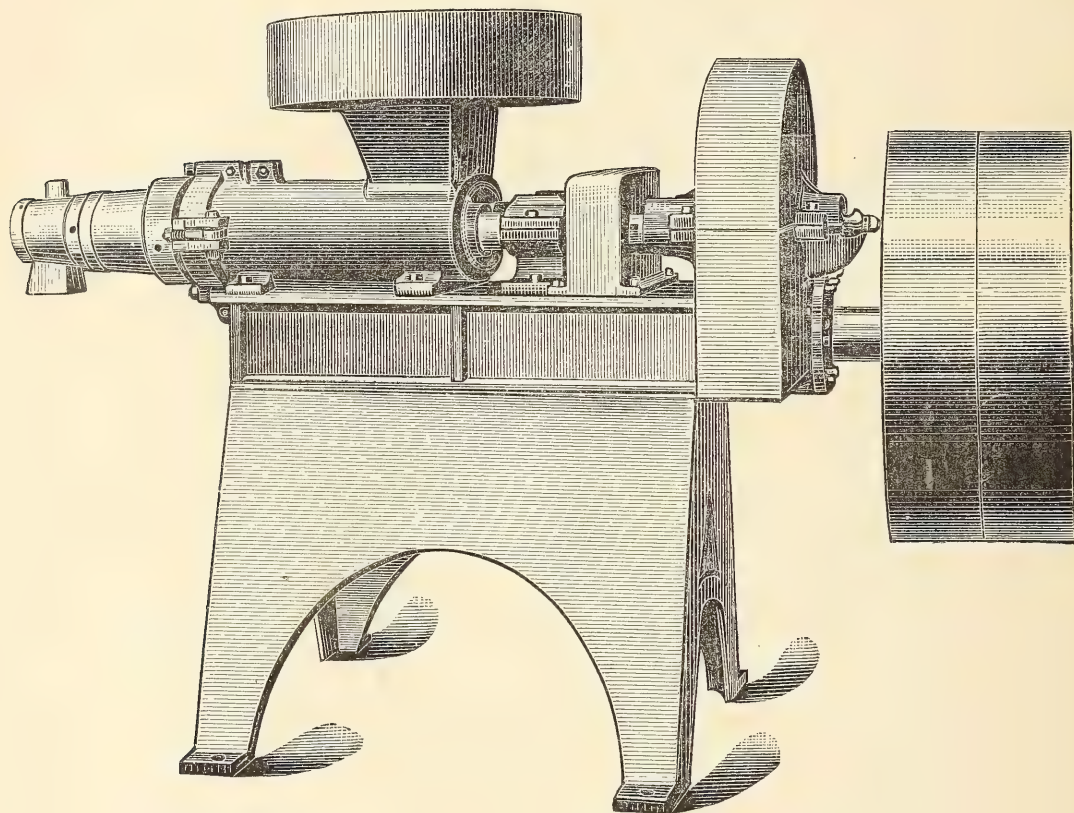
“obturateur” placed on the aperture, any heating of the perfumed mass of shavings is impossible even with uninterrupted working. The manipulation of changing colors and the mass of soap-shavings and the entire mechanism of this plodder is simplified as much as possible ; it has a capacity of 450 pounds per hour. It is furnished either with cone-pulley or fast and loose pulley.

The “Compound Helix Continuous Plodder,” patented and



manufactured by Rutschman Bros., of Philadelphia, is shown in Fig. 92. The improvement consists in the feature whereby soap comes continuously from the machine, a variation from the usual style of charging a cylinder with soap and compressing the mate-

Fig. 92.



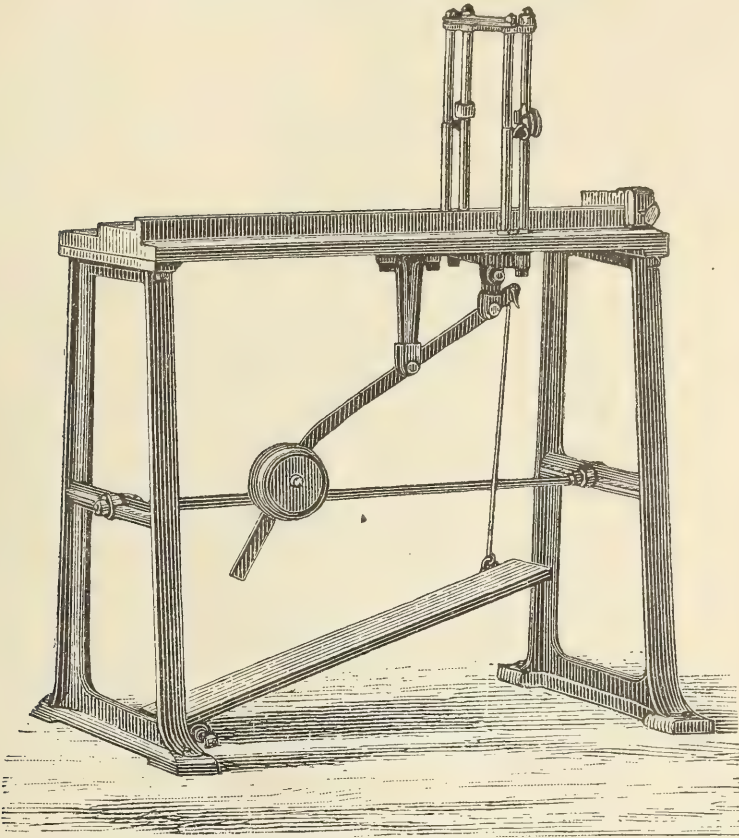
Compound Helix Continuous Plodder.

rials by means of the hydraulic—a screw or other pressure—in order to make the soap solid before it can be run into a bar. The soap is charged in the hopper and comes continuously from the opening in front of the machine (in a bar of any shape desired) perfectly solid and entirely free from streaks or other defects. The soap, as soon as it passes through the continuous plodder, can be cut and pressed at once without placing on racks to dry. The construction of the machine is so simple that skilled labor is unnecessary to operate it. It has a capacity of 250 to 350 pounds per hour, the power required being five horse-power.

*Soap-cutting machine.*—Where the plodder is not provided with a cutting apparatus, a special machine for the purpose is required.

These machines are of various sizes, and being entirely constructed of iron, are very durable. The working table (Fig. 93) is smoothly planed. The horizontal wire moves up and down upon two small vertical columns by treading upon the treadle

Fig. 93.



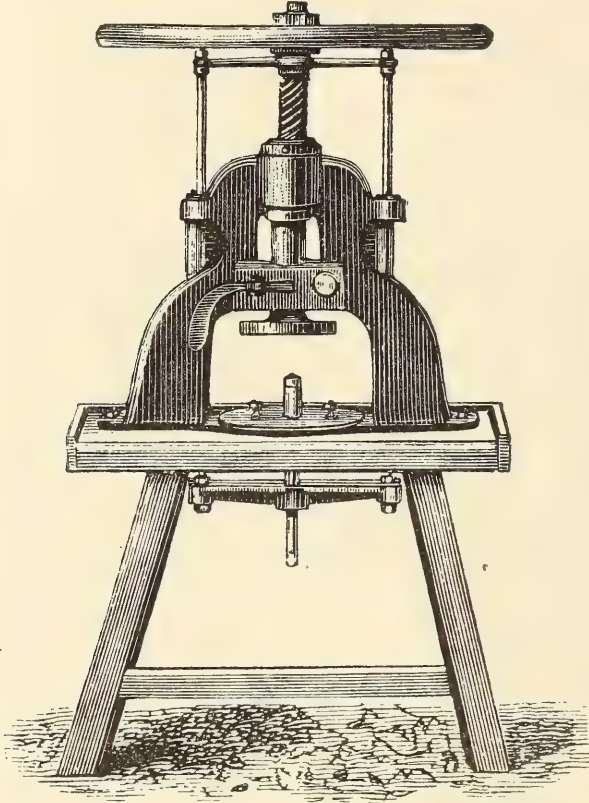
Cutting Machine.

under the table, the counter-weight again raising the wire. A shiftable back-square determines the length of the cakes. The machine being worked by a treadle, both hands are left free, the left pushing the bar forward, while the right removes the cakes. This machine requires but little force, so that it can be run by a girl.

Before pressing, the cakes must be heated to facilitate the work and obtain a fine impression, and after pressing the cakes have to be dried. This is effected in a drying-room heated either by a stove or hot air (see page 282).

*Soap-presses.*—The cakes being sufficiently dry, it is frequently necessary to give them finish, which is often done in a hand-press in a plain mould; but occasionally a mould-box with hinged

Fig. 94.



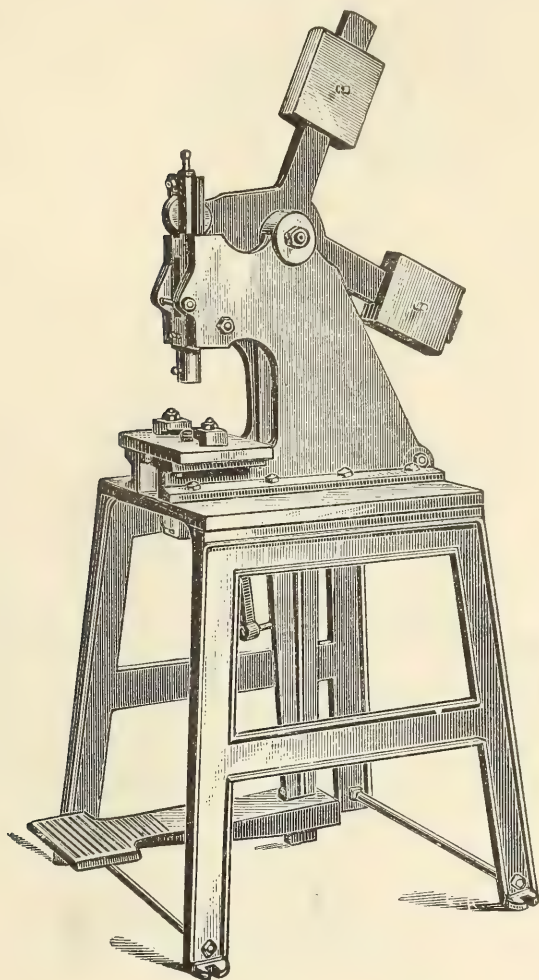
sides is employed, with a screw-press such as shown in Fig. 94. For larger tablets a foot-power press is desirable. Fig. 95 represents a foot-power press manufactured by Messrs. Rutschman Bros., of Philadelphia. It is worked by a treadle, leaving the hands free to handle the soap. It has adjustable balances to vary the blow. It works easily and can be operated by a boy. It is adapted from the smallest size toilet-soap to a two-pound laundry soap.

The soap stamping-press, shown together with box and dies in Fig. 96, is manufactured by Alfred W. Houchin, of Brooklyn. This press is adapted for stamping any sized cake of soap, is simple in construction, and adjusted to any sized dies. It has a vertical treadle motion, is easily worked, and gives a direct blow



to the soap. The box and dies for stamping soap are made of hard brass, and consist of an upper and lower die fitted to a box,

Fig. 95.

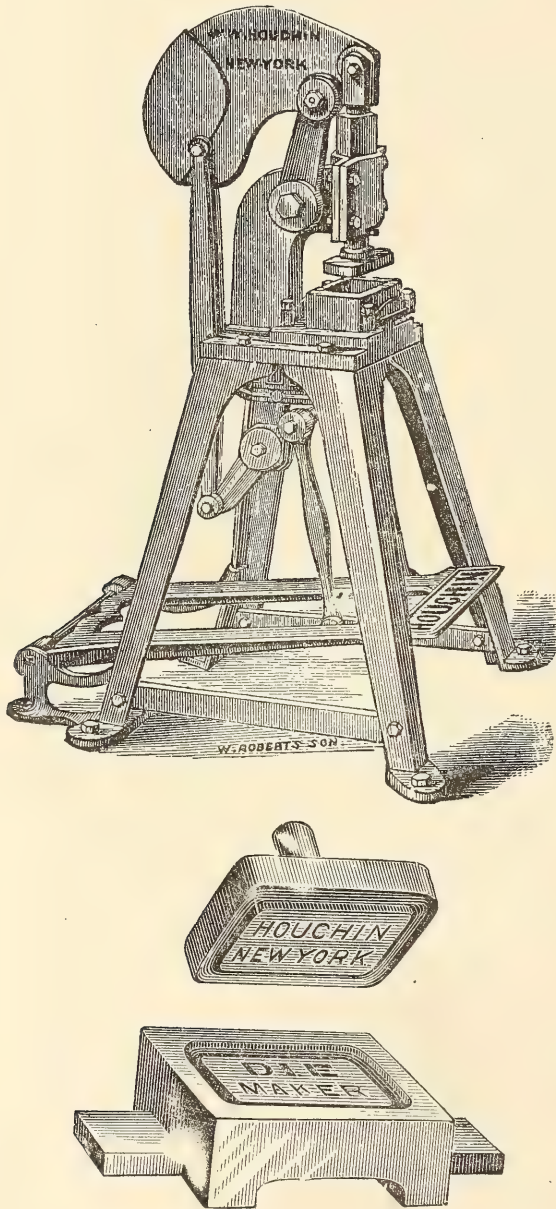


which is clamped to the bed of the press, with the bottom die in the box, the top die being fastened to the plunger. The dies can be engraved of any desired size and shape to suit, or can be made with interchangeable plates for the engraving. By this means, different brands of soap can be pressed with the same die by only changing the plate.

The improved swing foot-level soap-press, manufactured by H. W. Dopp & Son, of Buffalo, N. Y., is shown in Fig. 97. The slide is provided with two arms, one extending on each side. To these are attached adjustable uprights, *b b*, which rest on both

ends of the male die to prevent its warping. The press is provided with three lifting bolts, to push the soap perpendicularly

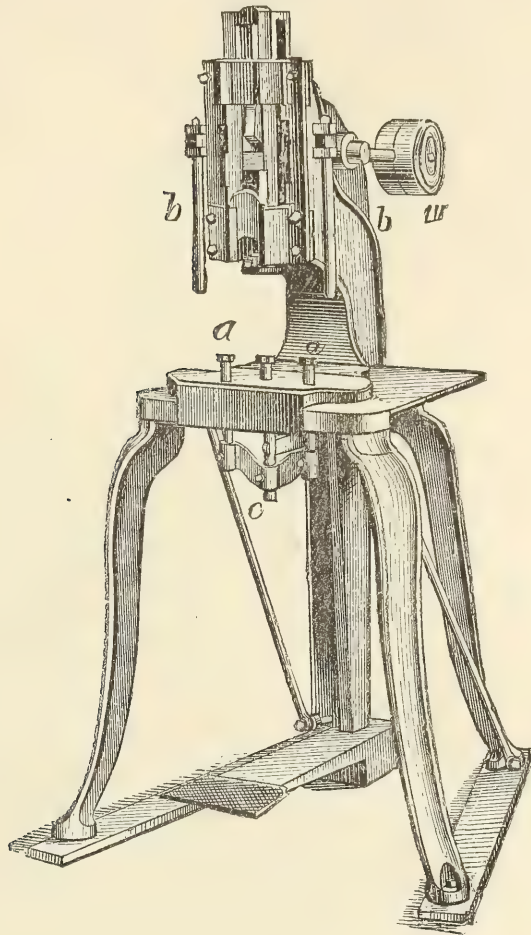
Fig. 96.



up and out of the mould box. The side bolts *a a* are connected with the centre bolt by means of a cross head *c* and set screws. When desired to use the press for ordinary work, the uprights *b b* and the cross-head may be taken off. This press can be used

for pressing a bar of soap from a few ounces up to 3 or 4 pounds in weight and 14 inches long.

Fig. 97.

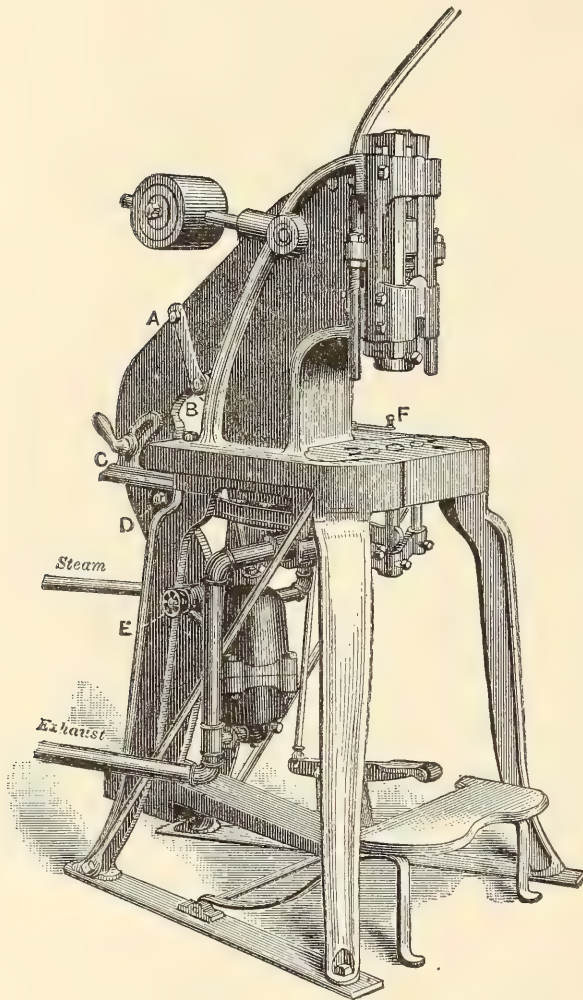


Similar in design is the steam soap-press, shown in Fig. 98, which is also manufactured by H. W. Dopp & Son, of Buffalo, N. Y. It has a single-acting steam-cylinder placed underneath the bed in such a position that its piston, by means of a roller attached to the end of the piston-rod, acts upon a cam surface of the swing or pendulum-lever, as indicated at *B*. A hook, *A*, attached to the piston-rod engages with a stud on the swing or pendulum-lever, and prevents the latter from recoiling after having returned from giving the blow, as it cannot fly back without pulling out the piston. Thus the unpleasant and dangerous vibration of the upper die-block is prevented. The



steam-supply pipe enters a governor or regulator which can be set by hand-wheel *E*, so that the press gives a blow of required

Fig. 98.



force. When this has once been set, the press cannot give a stronger blow than that for which it is set, no matter how much steam pressure the boiler may supply. To the right of this governor, *E*, is shown a balanced valve steam-trap which drains off all condensed water, and insures the admission of dry steam only to the cylinder, no matter how far the press may be from the boiler. The admission of steam is controlled by a foot-treadle, shown at the right of the cut, the heel of the foot resting on the foot-rest. The handle *F* serves to control the

exhaust in such a manner that the pendulum-lever returns with just enough force to eject the pressed soap, and no more. This ejection of the soap is accomplished by a cam, *C*, which is pivoted at one end to the pendulum-lever, and clamped to the latter by a jam-nut and arcs. Against this cam works, by means of a roller, a lever which with its other end actuates the centre lifting bolt. By unclamping this cam, shifting it up or down, and reclamping, the height to which the soap is lifted is regulated. This arrangement lifts the soap so gradually that there is no danger of throwing the cake of soap out against the upper die-block, and defacing the impression, no matter how fast the press is worked.

By throwing back hook *A*, and raising the foot-rest, the press is at once transformed into an ordinary foot-press. This is a great convenience in setting the die, as well as furnishing means of working when there is no steam.

*Finishing and polishing the soap-cakes.*—The cakes of soap, after they are pressed, stamped, and apparently ready for market, are often dried before being packed. In this drying they may lose some of their lustre. The remedy formerly used for this was to scrape the cakes off, and rub them with a woollen cloth dipped in strong alcohol.

This somewhat troublesome process has been superseded, according to Dupuis, by the method of exposing the soap before or after drying to a current of steam. The steam can be perfumed with any fragrant odor by passing it, before reaching the soap, through a cloth impregnated with fragrant materials. The steam causes at once a change upon the surface of the bars or cakes of soap, and forms, according to the fats used in their preparation, either a super-palmitate or super-stearic palmitin soda combination. If this operation is carefully done it closes up all the pores and uneven spots and, when dry, forms a very lustrous coating, which does not suffer, even under the moulding-press. No other method of polishing will give such a beautiful, even, and lustrous coating as that caused by steaming. Further advantages of this mode of operating are economy in time, manual labor, and prevention of all loss. It will especially preserve the soaps in damp storehouses, on sea voyages, and in the show-windows of stores, where they are exposed to the rays of the sun.

*Coloring toilet-soaps.*—With the many other improvements in the manufacture of toilet-soaps, there has been a corresponding advance in the character and nature of the coloring so necessary to their attractive appearance. While in former times ochres, chromes, and metallic oxides of iron, as siennas, umbers, etc., were used, with many other mineral substances, the object is now gained by other means, chief among which are the aniline colors.

There are, however, necessary but a few primitive colors, as almost any shade can be produced by a suitable blending of them. Thus yellow and orange are made with the naphthaline-yellow or cadmium-yellow; red is still made with vermilion; blue with ultramarine; green with Guinet's green, which is the borate of chrome; browns are made with sugar color, cutch, and chocolate modified with red or yellow. There are also constantly occurring many colors used in dyeing that may find application for soaps, which can, however, only be known by experiment.

Of the aniline colors, fuchsin, parmeanilin, eosin, Martius yellow, Bismarck brown, etc., are used.

In coloring soaps it is generally most desirable to color during the process of grinding in the mill, as it has several advantages. It prevents the color from being injured or altered by the heat; it gives the full brilliancy expected from the substance and with much less trouble. Many of the cheaper soaps being cut from the solid and not subjected to the milling and plotting processes, have to be colored and perfumed in the kettle or in the frame.

*Stock-soaps for milled toilet-soaps.*—The stock-soaps are generally prepared from 9 parts of fresh tallow free from odor and 1 part of cocoanut-oil, the addition of the latter securing a more abundant lather. Frequently a larger proportion of cocoanut-oil is used, but the above composition yields the most durable soaps.

Suppose 2000 pounds of fat are to be converted into stock-soap, the combination would therefore consist of 1800 pounds of tallow and 200 pounds of cocoanut-oil. The fats, which, as previously mentioned, must be entirely pure and fresh, are washed before boiling with salt water. Combination is next introduced with caustic soda lye of  $10^{\circ}$  B., and the addition of this lye continued until there are about 1200 pounds of it in the kettle. After the establishment of a thorough combination, boiling is con-



tinued with lye of  $25^{\circ}$  B., of which about 1400 to 1500 pounds are required. Thickening of the soap has to be prevented by a timely addition of lye. After fitting the soap to a slight touch it is salted out, about 10 per cent. of salt being required for the purpose. The soap is then allowed to stand over-night and the next morning the sub-lye is pumped out or drawn off. Sufficient pure water to reconvert the soap to a paste is then brought into the kettle; the soap is then again fitted to a slight touch with lye of  $25^{\circ}$  B., salted out, and the sub-lye allowed to subside by standing over-night. The next morning the sub-lye is again removed and fresh lye of  $20^{\circ}$  B. brought into the kettle, the quantity depending on the lower width of the kettle, though it must be sufficient for the soap to float upon it and prevent it from scorching. The soap is then boiled clear in the same manner as given for curd-soaps. The next morning the lye is again removed and the soap ground with water or weak lye until it flutters when thrown with the spatula and the sub-lye begins to become pasty. After standing at least 36 hours in the covered kettle, the soap is brought into the frames and somewhat crutched to promote uniform congealing.

For a *palm-soap for stock-soap* and for toilet-soap we refer to the formulas previously given for palm-soaps, and for this purpose advise extra care in selecting the materials.

*Cocoanut-oil soap* for toilet purposes should have a different manipulation. The oil not being saponifiable in weak lyes, it is always necessary to use lyes of  $28^{\circ}$  to  $36^{\circ}$  B. They need not be entirely caustic, as this oil can also be saponified with carbonated lye, though it takes, of course, more of it and requires a longer time. For this soap it is now customary to use a portion of lard, cotton-seed oil, or any other white grease or oil, but the cocoanut-oil for toilet-soaps should be the best Cochin China oil. By using a certain proportion of potash lye the soap retains a more plastic consistency and is much improved.

*White soap from cocoanut-oil.*—To prepare 400 pounds of this soap, bring into the kettle 200 pounds of pure white cocoanut-oil and add afterwards 200 pounds of colorless and perfectly limpid lye of  $30^{\circ}$  B.

All being ready, heat the kettle and, to accelerate the combina-

tion of the substances, stir well from time to time. Under the influence of heat the material, which at first was in the form of grains, softens and liquefies. Continue to heat slowly and gradually until the combination between the oil and the alkali is effected, which generally takes place when ebullition begins.

When properly made the soap has the appearance of a fluid, homogeneous, and syrupy paste. Its color is amber white. It is useless to boil it. Stop off the heat and draw off the soap into the frame.

If, on the contrary, it happens, when the mixture begins to boil, that a certain quantity of oil swims on the surface, it may be combined with the saponified mass by adding 10 to 12 pounds of cocoanut-oil soap. The same result may be obtained by adding 8 to 10 quarts of pure water. After stirring a few minutes the homogeneity of the soap is re-established and the combination of the substances perfected. The heat is then stopped and the soap drawn off into the frame.

Obtained by the above process the soap is very white, does not contain any excess of alkali or oil, and may be employed for toilet-soaps. From the quantities indicated above, 396 to 420 pounds of soap are obtained, according to the quantity of water added. The operation lasts about one hour.

*Half palm-soap.*—Either of the following formulas may be used: I. White tallow 900 pounds, palm-oil 400, cocoanut-oil 200, yellow resin 100.

II. Tallow 700 pounds, palm-oil 300, cotton-seed oil 400, resin 200.

III. Lard 550 pounds, tallow-oil 400, cotton-seed oil 450, resin 200.

The proportions of these substances are not fixed, and vary according to the uses for which the soap is intended. In England this soap is prepared with common tallows and an addition of resin. In France, where it is used only for toilet soaps, it is better attended to, and its purification is more complete. The above formulas give a soap of superior quality, and the use of which is very advantageous in the preparation of toilet soaps. The palm-oil may be bleached or not, but must always be purified.

The process of preparing the soap is as follows: By a gentle heat, melt the tallow and oils in the kettle; pour in 100 gallons of new lye of  $8^{\circ}$  or  $10^{\circ}$  B.; heat slowly and gradually, stirring from time to time, and, when ebullition begins, moderate the action of the heat, to avoid a too rapid reaction in the mass. After continuing the ebullition for about four hours, pour little by little on the paste from 35 to 50 gallons of new lye of  $15^{\circ}$  to  $18^{\circ}$  B., and incorporate it by stirring for about fifteen minutes. This being done, continue to boil for three hours, or rather until the paste appears quite homogeneous, and has acquired a certain consistency. Then a new quantity of 35 gallons of lye of  $20^{\circ}$  B. may be added, and, after a new ebullition for two hours, the first operation is finished.

Pasting being finished, the heat is stopped off, and after a few hours' rest, pour into the kettle a limpid lye of coction, *i. e.*, salted lye of  $20^{\circ}$  to  $25^{\circ}$  B., or a new lye containing salt in solution. While one man pours in the lye, another stirs the paste all the time. When the quantity of salt lye introduced into the kettle is sufficient, the soap is transformed into small grains, and the lye separates abundantly. After resting five or six hours, draw off the lye. About two-thirds of the lyes which have been used are drawn off; they have a yellowish color and mark, when cold,  $15^{\circ}$  to  $16^{\circ}$  B. The pasty mass left in the kettle has a fine yellow color.

The clear boiling of this soap is very little different from pure palm-oil soap. Like the latter, it is effected with new and caustic lyes of soda of  $25^{\circ}$  or  $28^{\circ}$  B. When the operation is done in two services, lyes of  $18^{\circ}$  or  $20^{\circ}$  B. are used for the first service, and lyes of  $25^{\circ}$  or  $28^{\circ}$  B. for the second. When, on the contrary, clear boiling is finished in a single operation, lyes of  $25^{\circ}$  B. are used. This last process is the quickest and most economical.

The lyes being drawn off, pour into the kettle from 150 to 175 gallons of new lye of  $25^{\circ}$  B.; heat and give a gentle boiling, for in the first hours the soap dilates and swells considerably. Its surface is then covered with an abundant froth, which gradually disappears only as clear boiling progresses. It is necessary to stir from time to time during the whole of the operation. This



agitation is very important, for it accelerates the clear boiling of the soap. When the soap has been gently boiled for three or four hours, the heat may be increased without fear of scorching it. Generally after eight or ten hours of ebullition with lye of  $25^{\circ}$  B., the soap is completely boiled. The froth has entirely disappeared, or there remains very little on the surface of the soap, which then has the form of hard and dry grains. When these grains are pressed between the fingers, they form thin and hard scales. The resin has been added at the beginning of clear boiling, so as to saponify it completely. When the soap is sufficiently boiled, which is known by its forming scales, stop off the heat, let it rest a few hours, draw off the lye, and proceed to the fitting.

Two operations are necessary to completely refine the soap. The first has for its object to soften the grains of the soap, and precipitate the coloring and heterogeneous substances and the excess of caustic lye it contains.

When the lye has been drawn off, pour into the kettle 100 gallons of new lye of  $8^{\circ}$  or  $9^{\circ}$  B., and heat gradually until boiling, being careful to stir the mixture well. When the grains of soap have become soft, cease the stirring; and to complete the precipitation of the strong lye contained in the soap, boil for five or six hours, or even eight hours. As by such a long ebullition the grain of the soap has a tendency to be formed again, pour from time to time into the kettle a few pails of lye at  $2^{\circ}$  B., or even pure water. It is, however, necessary that the soap should be always separated from the lye; this is ascertained by pouring some of the soap into a glass, and, if so, the lye precipitates to the bottom of the glass. It is important and essential to have, during the whole operation, the lye separated from the soap, to obtain the separation of the strong lye mixed with the paste. When this result is obtained, stop off the heat, and cover the kettle. Let it rest six hours, then introduce the soap into a second kettle, and proceed to a second liquefaction.

However great the care taken in the first liquefaction, the soap has not been completely deprived of all its causticity; it always contains a certain quantity of caustic alkali which must be eliminated to obtain a pure product. This is the object of the

second liquefaction. But to obtain all the good results this operation may produce, substitute for the caustic lyes of soda-ash a *non-caustic solution of crystallized soda*. By its extreme purity and the absence of causticity, this solution completely purifies the soap, depriving it of all its caustic parts. Pour into the new kettle about 36 gallons of a solution of crystallized soda of  $4\frac{1}{2}^{\circ}$  or  $5^{\circ}$  B., and heat to a temperature near the boiling point. Then introduce the soap from the first kettle into the second, being careful not to draw off any sub-lye. This being done, boil the mixture gently for four or five hours, being careful to stir from time to time. By the ebullition with weak lyes (aqueous solution of crystallized soda), the soap entirely loses its granular appearance, and becomes syrupy, fluid, and homogeneous. As in the first liquefaction, a froth is formed on the surface of the soap, and this froth is more considerable on account of the greater dilatation of the paste. As by evaporation the lye concentrates, add from time to time very small portions of water, so as to keep the paste always fluid. The heterogeneous coloring and saline impurities will be precipitated by resting. The soap must not contain too much water, for in this case it would be too long in hardening. The signs by which it is ascertained that the paste is sufficiently liquefied are manifested by a slightly brackish coloration, which proves that the black soap has been precipitated to the bottom of the kettle, and is brought up in the mass by ebullition. When these characteristics are observed, the operation is finished; stop off the heat; cover the kettle, and let it rest eighteen or twenty hours. By resting, the black soap precipitates with the lye, and the pure soap is between it and the scum. After eighteen or twenty hours' rest, uncover the kettle, and remove the scum from the surface of the soap. Remove the pure soap, and bring it into a frame, passing it through a metallic wire-sieve. All the foreign substances in the soap remain on the sieve.

When all the pure soap has been brought into the frames, stir it well until it is cold; this manipulation is necessary to make it homogeneous. By operating as indicated the above quantities of fatty matters generally give:—

Soap scum	.	.	.	.	.	.	141 to 161 pounds.
Pure soap	.	.	.	.	.	.	2100 " 2160 "
Black soap	.	.	.	.	.	.	500 " 600 "

The scum and black soap are mixed in the next operation or used for a common soap. The half-palm soap has a very pure yellow color when manufactured with good materials. It has also a good odor and is useful for making many kinds of soap, such as honey, glycerin, marshmallow, etc.

The soaps here given may be called stock-soaps, for from them nearly all kinds of toilet-soaps can be formed by compounding different kinds in suitable proportions, milling, mixing, coloring, plotting, moulding, and perfuming to suit the kinds needed.

In the following we give a few formulas for milled soaps:—

*Savon de Guimauve (Marshmallow-soap).*—White tallow-soap and palm-oil soap each 40 pounds. Color with yellow ochre and orange mineral each  $\frac{1}{4}$  ounces, gamboge  $1\frac{1}{4}$  ounces, and perfume with oil of lavender 6 ounces, oils of peppermint and caraway each 2 ounces, oil of lemon  $1\frac{1}{4}$  ounces, and oils of thyme and rosemary each 10 drachms.

*Savon à la Rose.*—Tallow-soap 50 pounds, potato-flour 4 pounds. (The potato flour is used to give the soap the necessary consistency.) Color with 7 ounces of vermilion and perfume with rose-oil  $1\frac{1}{4}$  ounces, oil of geranium 8 ounces, essence of musk  $1\frac{1}{2}$  ounces, and essence of civet 1 ounce.

*Savon aux Fleurs d'Italie.*—White tallow-soap 40 pounds. Perfume with oil of citronella 3 ounces, oil of geranium 1 ounce, oil of verbena 2 ounces, oil of peppermint 5 drachms, and color brown with sugar color.

*Savon de Crimée.*—White curd-soap 64 pounds, palm-soap 16 pounds. Color with vermilion 10 drachms, brown ochre 4 ounces, ivory black 2 ounces, and perfume with oils of thyme, peppermint, and rosemary each 4 ounces, oil of lavender 10 drachms, oil of cloves 6 drachms, and tincture of benzoin 6 ounces.

*Savon de palme.*—Palm-soap and half-palm soap each 40 pounds. Perfume with oil of bergamot 8 ounces, oil of cloves 2 ounces, oils of cinnamon and lavender each 4 ounces.

*Savon à la violette.*—Stock-soap prepared from bleached palm-



oil 50 pounds, pulverized orris root 4 pounds. Color with *terra siena* 1 ounce, and perfume with bergamot-oil 10 ounces, oil of geranium  $2\frac{1}{4}$  ounces, oil of neroli 1 ounce, oil of lavender  $1\frac{1}{2}$  ounces, essences of civet and musk each 1 ounce.

*Elder-flower soap*.—Half-palm soap 200 pounds, dextrine 6 pounds. Perfume with oil of bergamot 1 pound, oil of lavender 4 ounces, oil of thyme 4 ounces, oil of cloves 2 ounces, and oils of cassia and almonds each 1 ounce, and color light green.

*Lemon-soap*.—White soap 100 pounds, starch 4 pounds. Perfume with oil of lemon 8 ounces, oils of bergamot and lemon-grass each 4 ounces, oil of cloves 2 ounces, and color yellow with cadmium yellow.

*Orange-soap*.—White soap 100 pounds, starch 4 pounds. Perfume with oil of orange-peel 16 ounces, oil of cinnamon 1 ounce, and oil of thyme 4 ounces, and color dark yellow with naphthaline yellow.

*Patchouli-soap*.—White stock-soap 50 pounds, potato-flour 4 pounds. Color with  $1\frac{1}{4}$  ounces of Cassel brown, and perfume with geranium-oil  $3\frac{1}{2}$  ounces, oil of patchouli 7 ounces.

*Heliotrope-soap*.—White curd-soap 80 pounds, palm-soap 20 pounds, starch 4 pounds. Perfume with oil of rosemary 4 ounces, oil of thyme 2 ounces, oil of rose-geranium 3 ounces, oil of cloves 1 ounce, balsam of Peru 3 ounces, and color light purple with a red and blue color.

*Frangipanni-soap*.—Palm-soap 30 pounds, white soap 20 pounds, dextrine 2 pounds. Perfume with oil of bergamot 4 ounces, oils of neroli and santal each 2 ounces, tinctures of vanilla and civet each 8 ounces. Color light brown with tincture of catechu.

*Cold-cream soap*.—White soap 30 pounds, spermaceti-soap 20 pounds, oil of almond  $\frac{1}{2}$  pound, caustic potash of 6° B. 1 pound, gum tragacanth 2 ounces.

Strip up the two soaps, place them in the hopper of the mill, dissolve the gum by previous soaking in a little water, mix with the oil and lye to a uniform consistency, then stir in the soap and grind in the mill until thoroughly combined. Care should be taken to have the mass as white as possible. Perfume the above

with oil of bergamot 5 ounces, oils of cloves and nutmeg each 1 ounce, oil of thyme 2 ounces, oil of bitter almonds 1 ounce.

*Savon de riz.*—54 pounds of wax-soap and 8 pounds of starch. Perfume with oil of geranium 1 ounce, oil of orange (Portugal)  $1\frac{1}{2}$  ounces, oil of bergamot  $1\frac{1}{4}$  ounces, oil of mirbane 1 ounce, tincture of benzoin  $\frac{1}{4}$  ounce. The soap remains white or is colored red with  $2\frac{1}{2}$  ounces of vermilion.

*Savon au bouquet.*—White stock-soap 30 pounds. Perfume with bergamot-oil  $1\frac{1}{2}$  ounces, oil of sassafras  $\frac{1}{2}$  ounce, oil of thyme 6 drachms, oils of lavender and cloves each 5 drachms, oil of geranium 2 drachms. Color yellow, brown, red, or green.

*Herb-soap (Dr. Borchardt's).*—White stock-soap and olive-oil soap each 60 pounds, starch 3 pounds. Perfume with oils of cassia and lavender each 1 pound, and oil of bergamot 2 pounds.

*Lily-soap.*—Wax-soap 60 pounds, starch 6 pounds. Perfume with oil of bergamot 5 ounces, oil of geranium 2 ounces, oil of cassia  $\frac{1}{2}$  ounce, oil of santal 3 drachms, oil of cedar, tincture of musk, and tincture of tonka-bean each 1 ounce, tincture of storax 3 ounces.

*Superfine toilet-soaps.*—In the following we give some formulas for fine and superfine soaps, to which it is recommended to add a little wax, which will give to the soaps consistency and smoothness and improve their quality.

*Ambergris-soap.*—Grease perfumed with ambergris and musk 25 pounds, jasmine pomade of flowers No. 24 and rose pomade of flowers No. 24 each 10 pounds, beeswax 1 pound, gum tragacanth 3 ounces, caustic soda lye of  $33^{\circ}$  B. 25 pounds. Color light brown with sugar color.

This soap is made of select materials by the cold process, and after being made is allowed a few days to dry before milling; the musk and ambergris have to be added to the grease some weeks before, frequently melting and stirring.

*Benzoin-soap.*—Lard with benzoin 30 pounds, cocoanut-oil and tallow, each 10 pounds, soda lye of  $35^{\circ}$  B. 26 pounds, gum tragacanth 2 ounces. Perfume with oil of bergamot 8 ounces, oil of lavender 3 ounces, oil of pimento 1 ounce, flowers of benzoin and tincture of benzoin, each 3 ounces.

Saponify in the usual way. The lard with benzoin is made by

infusing the lard with the powdered gum (2 ounces to the pound) for a month, occasionally melting and stirring. Melt and strain off the clear lard before using.

*Jonquille-soap (superfine).*—Orange-flower pomade No. 24 20 pounds, tuberose pomade No. 24, jasmine pomade No. 24, and castor-oil, each 10 pounds, white wax  $1\frac{1}{2}$  pounds, gum tragacanth 2 ounces, caustic soda lye of  $36^{\circ}$  B. 27 pounds.

Saponify as carefully as possible, avoiding too much heat. The soap will be a light yellow. To enhance the color add a little anatoline.

*Millefleur-soap.*—Lard with vanilla 20 pounds, lard with ambergris, rose pomade (aux fleurs) No. 24, and butter of cocoa, each 10 pounds, chocolate 2 pounds, caustic lye of  $36^{\circ}$  B. 26 pounds. Perfume with oil of orange (Portugal) 8 ounces, oil of lavender 4 ounces, oil of cloves 2 ounces, oil of nutmegs 1 ounce, tincture of musk 4 ounces.

The chocolate will give the proper color. Operate with care and you will have a fine soap.

*Savon à la Marechale (surfin).*—Lard with musk and lard with ambrette, each 10 pounds, pomade (aux fleurs) No. 24 of cassia, jasmine, and rose, of each 10 pounds, olive-oil 1 pound, white wax 2 pounds, gum tragacanth 2 ounces, caustic lye of  $36^{\circ}$  B. 28 pounds.

Saponify carefully and color with a little sugar color.

*Savon hygiénique (extra fine).*—Orange-flower pomade No. 24 10 pounds, rose pomade No. 24 5 pounds, palm-oil (bleached) 20 pounds, cocoa butter 5 pounds, olive-oil 10 pounds, white wax 1 pound, caustic lye of  $38^{\circ}$  B. 24 pounds, gum tragacanth 2 ounces. Perfume with oils of santal and geranium, each 2 ounces, oils of valerian and melisse, each 1 ounce, oil of orange 4 ounces, oil of thyme 2 ounces.

Avoid too much color; the soap should have a yellowish-brown that needs no addition.

*Savon à la violette de Parme.*—Violette Pomade No. 24 20 pounds, rose pomade No. 24 and cassia pomade No. 24, each 10 pounds, bleached palm-oil 10 pounds, soda lye of  $36^{\circ}$  B. 25 pounds, gum tragacanth 2 ounces.

Give it a purple color, not too dark.



*Lettuce-soap.*—Lard with lettuce 20 pounds, cassia pomade No. 24 10 pounds, spermaceti 5 pounds, castor-oil 5 pounds, bleached palm-oil 10 pounds, caustic lye of  $36^{\circ}$  B. 26 pounds, gum tragacanth 3 ounces. Perfume with oil of bergamot 6 ounces, oil of thyme 2 ounces, oils of valerian and cloves, each 1 ounce.

Color light green with Guinet's green. The lard with lettuce is made by melting the lard with its own weight of lettuce leaves, keeping it at the melting-point for some hours, or until the leaves have parted with their color and juice ; then strain off for use.

*Cucumber-soap.*—Operate as for lettuce-soap, using the fruit.

*Rose-leaf soap (extra fine).*—Rose pomade (aux fleurs) No. 24 and lard, each 20 pounds, cocoanut-oil 10 pounds, white wax 2 pounds, soda lye of  $36^{\circ}$  B. 20 pounds, potash lye of  $30^{\circ}$  B. 12 pounds, gum tragacanth 3 ounces. Perfume with oils of roses, geranium, and bergamot, each 2 ounces, oil of rhodium 1 ounce, oil of cinnamon  $\frac{1}{2}$  ounce, and color light pink with aniline (fast red).

*Shaving-soaps.*—These soaps are prepared in various ways. The properties demanded from a good shaving-soap are, that it yield a good and strong lather, which should remain standing for a long time and at the same time be mild and delicate.

To combine these properties fats yielding a good lather have to be chosen and saponified with alkalies exerting a mild effect upon the beard and skin. Hence potash soaps are better adapted for the purpose than soda soaps, and of the latter those containing cocoanut-oil are more suitable than those without it. To obtain a shaving-soap answering all reasonable demands proceed as follows :—

Saponify 100 pounds of tallow with lye of  $15^{\circ}$  B. and boil clear. After removing the salty lye add 15 pounds of cocoanut-oil and saponify it with potash lye of  $25^{\circ}$  B. When the whole is thoroughly combined, fit the soap slightly, and after allowing it to boil through, bring it into the frame. Perfume with  $3\frac{1}{2}$  ounces of oil of cassia and  $4\frac{1}{4}$  ounces of oil of thyme.

*Shaving-soap by the cold process.*—To obtain shaving-soap possessing all the good properties desired it is generally prepared by boiling. It being, however, not always desirable to prepare large

quantities at one time, the following process, which will give a good shaving-soap in a short time, is recommended :—

Bring into the kettle 80 pounds of white tallow and 40 pounds of cocoanut-oil. Heat to about  $99.5^{\circ}$  F., and the fats being melted, add in the usual manner 64 pounds of caustic soda lye of  $30^{\circ}$  B. and 16 pounds of potash lye of  $30^{\circ}$  B., until the mass forms a well-combined, homogeneous paste. The entire operation requires at the utmost fifteen to twenty minutes. It is finished when the surface of the soap becomes covered with a film which constantly reforms, notwithstanding stirring. Perfume the soap with oils of lavender and thyme, each  $3\frac{1}{2}$  ounces, oil of cumin 7 ounces, oil of bergamot  $10\frac{1}{2}$  ounces. The perfume is added to the soap, with constant stirring, before bringing it into the frame.

*Paris transparent shaving soap.*—Saponify 20 pounds of castor-oil and 10 pounds of Cochin China cocoanut-oil with 52 to 54 pounds of potash lye of  $20^{\circ}$  B., and boil like soft soap, until the mass is short, thick, and clear. After cooling to  $167^{\circ}$  F., add 6 pounds of glycerin, crutch thoroughly through, and then stir in 3 pounds of 90 to 92 per cent. alcohol. Should the soap lose transparency, add water until it is again clear and transparent. Perfume the soap (calculated for 100 pounds) with lavender-oil  $5\frac{1}{4}$  ounces, oil of cumin  $3\frac{3}{4}$  ounces, and oils of cassia and bergamot, each 1 ounce.

*Shaving-soap by the warm method.*—A good shaving-soap is obtained by saponifying 200 pounds of tallow with lye of  $15^{\circ}$  B., consisting of 4 parts soda lye and 1 part potash lye, adding the soda lye first and then the potash lye. When the paste is clear, add 40 pounds of cocoanut-oil, together with 15 to 20 pounds of potash lye of  $20^{\circ}$  B., and salt out, so that the grain appears like ground. Then bring the soap into small frames, and after perfuming it with a few drachms of lavender-oil, add some hot water, and crutch until the soap is cold.

*English shaving soap.*—Pure white tallow, freshly rendered, 40 pounds, Cochin China cocoanut-oil 20 pounds, lye of  $30^{\circ}$  B., prepared from crystallized soda, 38 pounds; potash lye of  $30^{\circ}$  B. 8 pounds. Melt the tallow, and allow the cocoanut-oil to dissolve in it. When all is melted, heat the fat to  $144.5^{\circ}$  F., stir in the lye, heat again to  $144.5^{\circ}$  F., and perfume with oil of

bergamot  $1\frac{1}{2}$  ounces, oil of cumin 2 ounces, oil of Portugal 5 drachms, and oils of thyme and lavender, each 11 drachms.

Mix the oils, and add the mixture to the soap before bringing it into the frames.

The soap prepared in the above manner has not only a pleasing appearance, but does not contain an excess of alkali, and is, therefore, not only suitable for shaving, but also for toilet use.

*Windsor soap for shaving.*—Pure white tallow 80 pounds, Cochin China cocoanut-oil 40 pounds, soda lye of  $30^{\circ}$  B. 68 pounds, potash lye of  $30^{\circ}$  B. 12 pounds. Perfume with oil of bergamot  $5\frac{1}{4}$  ounces, oil of cumin 13 ounces, oil of rosemary 3 ounces, and oil of lavender 3 ounces.

*Soft toilet soaps.*—The alkaline base of these soaps is potash, and the fatty substance generally used consists of good lard, though sometimes 10 to 20 per cent. of cocoanut-oil is introduced to promote the lathering properties of the soap. The soaps which are generally perfumed with oil of bitter almonds, and are known as almond soap-cream, etc., serve chiefly for toilet purposes and shaving, and must, therefore, be very delicate and mild.

Prepare first a clear caustic potash lye of  $20^{\circ}$  to  $21^{\circ}$  B. Now bring 50 pounds of white lard and 10 pounds of cocoanut-oil into a clean, roomy kettle; apply gentle heat, and, when the fat is melted, add 50 pounds of potash lye of  $20^{\circ}$  to  $21^{\circ}$  B.; keep the mixture at a temperature of from  $167^{\circ}$  to  $189^{\circ}$  F., and stir constantly. By the action of the heat and stirring, the aqueous portion of the lye is evaporated, and the mixture acquires a thicker consistency. It may happen that a portion of the fatty substances separates from the mass, which is chiefly the case when the temperature of the mixture is raised to a degree approaching the boiling point, because at this temperature concentrated lyes, with but few exceptions, have, as a rule, little affinity for the fatty substances. It may, however, also be due to the inadequacy of alkali in the mixture. In the first case, the combination is restored by moderating the heat, and, in the other, saponification is completed by pouring in a quantity of stronger lye.



For the first operation, which is called preparatory boiling, about four hours are required. For the complete saponification of the fat, about 30 pounds of potash lye of  $35^{\circ}$  B. are then gradually added, care being had to keep the mixture homogeneous by constant stirring, and keeping the temperature below the boiling point and as stationary as possible between  $167^{\circ}$  and  $189^{\circ}$  F. The end of saponification is recognized by the paste acquiring a very thick consistency. Stirring now becoming more difficult, the fire is removed, as, notwithstanding the stirring, the soap might settle on the bottom, and scorch, which would not only change the quality of the soap, but destroy its white color. Many manufacturers prepare this soap in an iron kettle, with double bottom heated by steam; some use silver kettles, which are preferable, because the soap will retain in them all its whiteness.

Fig. 99 represents a kettle with a double bottom heated by steam. This kettle is of tinned copper, and may be used also to purify tallow and greases.

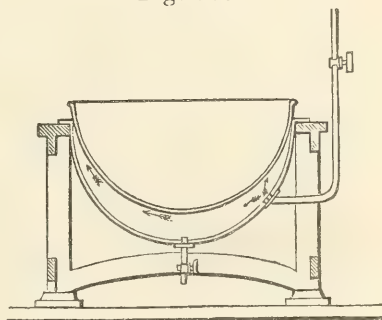
The entire operation of preparing this soft soap lasts from seven to eight hours. When the soap is cool, it has a soft and pasty consistency, and is poured into large stone or porcelain jars in which it is kept for use.

Soft soap, as obtained by the saponification of fatty substances by potash, has not that bright and nacreous appearance required for the toilet. To attain this state, it is ground in a marble mortar, and aromatized with oil of bitter almonds, about 1 ounce being sufficient for the above quantity.

Although oil of bitter almonds is principally used as a perfume for these *soap creams*, other fragrant substances are occasionally employed—for instance, liquid storax and benzoin, oil of cocoa, etc.

When the soap is required to be of a delicate rose-color, from 15 to 30 grains of vermilion to each pound of soap must be added and well incorporated with the pestle.

Fig. 99.



In the following we give a few formulas for various soft toilet-soaps:—

*Almond shaving cream.*—Take a few pounds of the above soap, introduce it into a marble mortar, and strongly triturate it with a wooden pestle. The operation is finished when the soap forms a soft and homogeneous paste; the more it is beaten, the finer it will become. To perfume it, incorporate from  $1\frac{1}{2}$  to 2 drachms of oil of bitter almonds per pound.

*Rose shaving cream.*—Add to the above soap, while triturating it in the mortar, one-half drachm of vermilion per pound, and perfume with attar of roses.

*Ambrosial shaving cream.*—Perfume with liquid storax and benzoin, oils of bergamot and cloves, and color purple with tincture of archil.

*Naples shaving cream.*—Boiled soft soap 50 pounds, gum tragacanth 2 ounces, tincture of musk 2 ounces, tincture of ambergris 1 ounce, tincture of balsam of Peru 3 ounces, oil of geranium 2 ounces; color light brown.

*Shaving-cream.*—Melt 20 pounds of hog's lard in a water-bath at  $212^{\circ}$  F., add with constant stirring 5 pounds of potash lye of 1.33 specific gravity, and keep the mixture at this temperature, gradually adding with constant stirring 5 pounds more of potash lye. Saponification is finished in a few hours. The cooled soap is made into a perfectly homogeneous paste by rubbing in a mortar, and for *crème d'amandes* perfumed with oil of bitter almonds, and for *crème de rose* with oil of rose, and in the latter case colored rose-red.

*Rypophagon-soap* is a mixture of equal parts of resin-soap and fine white soap perfumed with oils of aniseed and citronella.

*Crème d'ambrosie* is prepared from lard colored intensely red with alcanna, the finished soap showing a peculiar violet color. It is perfumed with oil of peppermint.

*Liquid glycerin-soap.*—Mix in a suitable vessel 100 parts of oleic acid with 314 parts of glycerin. Heat the mixture and compound it with constant stirring with 56 parts of potash lye of 1.34 specific gravity, whereby saponification is quickly finished. Now allow the soap to stand one to two days, then dilute it with an equal quantity of water, filter, and evaporate the filtrate to

half its quantity in a water-bath. To restore the soap to its thickly-fluid, honey-like consistency, add 10 parts of purified potash dissolved in as little hot water as possible. Any desired perfume can be given with oil of neroli or another volatile oil.

In this class of soap we include also those which, mixed with water, form emulsions and are used as cosmetics; they consist chiefly of soft soap, generally almond cream (*crème d'amandes*) and fat-oil.

*Amandine*.—Mix  $2\frac{3}{4}$  ounces of white syrup and 11 drachms of almond soap-cream, and gradually add with constant stirring 7 pounds of oil of almond perfumed with 11 drachms each of oils of bitter almonds and bergamot and 5 drachms of oil of cloves.

The preparation requires considerable experience and exertion of strength, as towards the end of the operation stirring becomes very difficult on account of the increasing consistency. The fresher the oil the better the preparation.

*Olivine*.—Gum arabic  $1\frac{1}{4}$  ounces, honey 4 ounces, the yolks of 5 eggs, soft soap 2 ounces, olive-oil 2 pounds, oils of limes and bergamot each 11 drachms, oil of thyme  $1\frac{1}{2}$  drachms, oil of cloves 5 drachms, and oil of cassia  $1\frac{1}{2}$  drachms. Rub the gum and honey together, then add the yolks of eggs, and add the perfume in the same manner as given for amandine.

*Glycerin jelly*.—Mix 4 ounces of glycerin with  $2\frac{1}{2}$  ounces of soft soap and stir in, in the manner above described, 4 pounds of almond-oil perfumed with  $2\frac{1}{2}$  drachms of oil of thyme. In summer it is advisable to use 5 pounds of almond-oil.

*Soap-balls or savonettes*, often called wash-balls, once very much used, are made of any good hard soap, cut into squares and rounded in the hand with a brass tool until spherical. Mottled soap marbled with vermilion and ultramarine is the kind most used. The transparent soaps are also formed into balls and have a good appearance.

*Medicated soaps*.—Every soap-maker engaged in the preparation of medicated soaps is aware of the painstaking care and accuracy required to make soaps according to a physician's prescription, and although many manufacturers turn out an excellent product there are many so-called medicated soaps brought into commerce which do not deserve the name, since they fre-



quently contain not even a trace of efficacious substances. Again, we find other medicated soaps which are inoperative on account of ignorance as regards the decomposition of the chemical agents added to the soap. In many iodine soaps, for instance, a small addition of potassium iodide is found, but no sodium hyposulphite, which is absolutely necessary to render the iodine efficacious. And thus there are many other soaps, which from a want of sufficient chemical knowledge are not properly composed, and instead of having a soothing and healing effect in skin diseases promote the diseased state.

Medicated soaps are prepared either in the cold or warm way similar to milled soaps.

Attention has been recently directed to some medicated soaps prepared by Dr. P. G. Unna, of which the super-fat salicylic-soap and super-fat zinc salicylic-soap are the most interesting. These soaps prepared from the best salicylic acid are successfully used.

1. As a disinfecting soap in all fungoid affections of the skin.
2. As an auxiliary remedy in the form of simple ablutions, with as hot water as possible in severe, obstinate, and strongly itching cases of eczema.
3. In acne, partially to promote the removal of the diseased horny layer and to lay bare the follicle closed by it and partially to remove the black horny points of the comedones.

The composition of the

*Super-fat salicylic-soap* is as follows: Super-fat stock-soap 95 parts, salicylic acid 5 parts. The soap is yellowish white and quite soft, and inclined with repeated soaking and drying to become brittle, and should, therefore, be kept dry.

*Super-fat zinc salicylic-soap* is composed of 88 parts of stock-soap, 2 parts of zinc oxide, and 10 parts of salicylic acid. This soap is white, very hard, yields little lather with cold water, but an abundant one with hot water, with which it is directed to be used.

Other super-fat soaps prepared according to Dr. Unna's directions are:—

*Super-fat tar-soap* contains 5 per cent. of tar.

*Super-fat sulphur-soap* contains 10 per cent. of precipitated sulphur.

*Super-fat tar-sulphur soap* contains 5 per cent. each of tar and precipitated sulphur.

*Super-fat camphor-sulphur soap* with 5 per cent. of camphor and 10 per cent. of precipitated sulphur.

*Super-fat camphor-soap* with 5 per cent. of camphor.

*Super-fat borax-soap* with 5 per cent. of sodium borate.

*Super-fat iodine-soap* with 5 per cent. of potassium iodide.

*Super-fat naphthol-soap* with 5 per cent. of naphthol- $\beta$ .

*Super-fat naphthol-sulphur soap* with 5 per cent. each of naphthol- $\beta$  and precipitated sulphur.

*Other formulas for medicated soaps. Benzoin-soap.*—Cochin China cocoanut oil 16 pounds, soda lye of 40° B. 8 pounds, tincture of benzoin 21 ounces, pulverized sienna 2 $\frac{3}{4}$  ounces.

*Benzoin soap (in the cold way).*—Prepare first by the cold way a good non-caustic soda-soap from a mixture of cocoanut-oil and lard, strip the soap, sprinkle the shavings with the color, then pour the benzoin tincture over them, mix in the mill, press, dry, and mould the finished soap.

For benzoin-soap use : 100 pounds of soap prepared as above and tincture of benzoin 8 pounds. Color brown with sugar color.

Benzoin tincture is prepared by treating a good quality of pulverized benzoin with alcohol. Benzoin soap has an agreeable vanilla-like odor.

*Camphor soap (in the cold way).*—Cocoanut-oil 40 pounds, and lye of 38° to 40° B. 20 pounds, stirred together at 106° F., camphor 21 ounces, and oils of rosemary and caraway each 7 ounces. The camphor is dissolved in the oil. It may also be dissolved in alcohol and the solution crutched into the finished soap.

*Carbolic soap.*—Half-palm soap 20 pounds, starch 1 pound, carbolic acid (crystals) 1 ounce, oil of lavender 2 ounces, oil of cloves 1 ounce.

*Carbol-glycerin soap.*—Tallow, cocoanut-oil, and lye of 38° B. each 30 pounds, alcohol 15 pounds, liquid carbolic acid and glycerin each 12 pounds, sugar color  $\frac{1}{2}$  pound. Prepare in the same manner as glycerin soap.

*Iodine soap* is used for the preparation of so-called iodine baths as a remedy for eruptions of the skin. The soap is prepared in the cold way as follows; 20 pounds of cocoanut-oil are saponified in the usual manner with 10 pounds of caustic lye of  $40^{\circ}$  B. and a solution of 3 pounds of potassium iodide in 4 pounds of water is added.

*Sulphur-soap*.—Saponify 20 pounds of cocoanut-oil with 10 pounds of caustic soda lye of  $40^{\circ}$  B. and stir in 4 pounds of flowers of sulphur.

The soap may also be prepared in the following manner: Take any good hard soap, half-palm for instance, and melt carefully with dissolved starch and add about 12 per cent. of flowers of sulphur, color yellow with naphthaline yellow. Perfume to fancy.

*Tar-soap*.—Melt together 20 pounds of cocoanut-oil and 3 pounds of coal-tar, and saponify the mass with 11 pounds of caustic lye of  $11^{\circ}$  B. This soap is chiefly used for eruptions of the skin.

*Another tar-soap* is prepared from: Cocoanut-oil 20 pounds, tallow 10 pounds, juniper-tar 5 pounds, soda lye of  $40^{\circ}$  B. 15 pounds. First saponify the fats and then add the tar; perfume may be added, though fine perfume would be lost in the strong odor of the tar.

*Tannin-soap*.—Dissolve 15 pounds of good tallow soap in a water-bath, and stir into it 1 pound of tannic acid (to be had of any druggist) and sufficient starch so that the mass can be formed into suitable cakes.

*Vaseline tar-soap*.—Melt 20 pounds of cocoanut-oil and 3 pounds of tar and saponify the mass with 11 pounds of lye of  $40^{\circ}$  B. Then dissolve 2 pounds of yellow vaseline and stir this together with  $\frac{1}{2}$  pound of lukewarm water into the soap.

Thus the intelligent soap-maker can prepare any kind of medicated soap by using the proper proportions of the substances, taking care that the medicinal agent may not be injurious to the skin or the general health.

*Thymol-soap* with 3 to 5 per cent. of thymol.

*Croton-oil soap* with 2 per cent. of croton-oil.

*Castor-oil soap* with 20 per cent. of the oil with other fats.

*Becktonide of Mending*



*Petroleum-soap* with 20 per cent. of the petroleum added to the other fats before saponification.

*Paraffin soap*.—The wax to the amount of 10 per cent. is added to the fats before saponification.

*Creasote-soap* with 2 per cent. of creasote.

*Turpentine-soap* with 5 per cent. of oil of turpentine.

*Alum-soap* with 10 per cent. of finely powdered alum.

*Mercurial-soap* with 6 per cent. of mercurial ointment.

*Irish moss-soap* with 5 per cent. of Irish moss dissolved in a suitable quantity of water and strained.

*Bran-soap* with 10 to 20 per cent. of bran.

*Cornmeal-soap* with 10 to 20 per cent. of cornmeal.

*Oatmeal-soap* with 10 to 20 per cent. of oatmeal.

*Gall and scouring-soaps*.—A good gall-soap for washing fine silk stuffs should not be wanting in any household. Such soap is readily prepared requiring no special apparatus.

Cocoanut-oil 6 pounds, tallow 2 pounds, Venetian turpentine  $1\frac{1}{2}$  pounds are brought into a kettle and heated to  $106^{\circ}$  F. Then stir in 4 pounds of caustic soda lye of  $38^{\circ}$  B. previously mixed with  $1\frac{1}{2}$  pounds of ox-gall, bring the soap into the frame and cover it lightly.

*Gall-soap*.—Saponify in the ordinary manner 20 pounds of cocoanut-oil with 10 pounds of caustic soda lye of  $38^{\circ}$  to  $39^{\circ}$  B. and add 10 pounds of ox-gall. Color green and add  $1\frac{1}{2}$  ounces of oil of turpentine.

*Gall scouring soap*.—In 40 pounds of cocoanut oil heated to  $99.5^{\circ}$  F., dissolve  $4\frac{1}{4}$  ounces of ultramarine green and saponify in the ordinary manner with 21 pounds of soda lye of  $38^{\circ}$  B.

In the mean while weigh off 6 pounds of potash-lye of  $10^{\circ}$  B. and 8 pounds of salt water of  $12^{\circ}$  B., stir into this solution 6 pounds of ox-gall and add all with constant stirring to the soap. Then stir in 1 pound of oil of turpentine and 4 ounces of potassium bichromate dissolved in 5 ounces of hot water. Ladle the soap into the frame and cover lightly.

*Scouring soap*.—Melt 10 pounds of cocoanut-oil and 6 pounds of kidney-tallow, and saponify with 8 pounds of lye of  $40^{\circ}$  B. Color the soap green with  $1\frac{1}{2}$  ounces of ultramarine green, and stir into it 1 pound of ox-gall and  $\frac{1}{2}$  pound of oil of turpentine.

*Breslau scouring soap.*—Dissolve 2 pounds of white curd-soap cut into fine shavings in  $4\frac{1}{2}$  ounces of boiling water, and add  $3\frac{1}{2}$  ounces of alcohol and 2 ounces of spirit of sal ammoniac.

*Floating soap.*—For certain purposes—bathing, etc.—it may be of advantage to have a soap which will float. According to Hilgers, such soap can be prepared from every kind of curd-soap by inclosing a sufficient quantity of a substance specifically lighter than water to render the soap itself lighter than water. Hollow glass-balls or hollow substances of water-proof paper are suitable for the purpose, but the best materials are pine-bark, cork, etc., they being light, cheap, infrangible, and indifferent towards the action of water. A piece of bark, cork, etc., corresponding in shape to the exterior of the cake of soap, and perforated by several holes, is inclosed in the soap in moulding, an intimate union of the two surfaces being effected by the soap running through the holes in the cork.

Instead of inclosing such light substances, floating soap can also be prepared by the formation of actual hollow spaces, impressions of any desired shape being made for this purpose upon the inner surfaces of two pieces of soap, which are afterwards cemented together by means of liquid soap.

Floating soap can be prepared in a still more simple manner by stirring into hot ground-soap, while cooling, finely pulverized sodium bicarbonate. At a temperature of  $158^{\circ}$  F., the sodium bicarbonate is decomposed into soda and carbonic acid; and the latter, seeking an escape, renders the soap-mass porous and spongy, in which state it finally cools, its specific gravity being reduced by nearly double the increase in bulk.

For preparing floating soap on a large scale, indirect steam has to be used, and the kettle provided with a stirring apparatus. As a rule, fats yielding a good lather, such as cocoanut-oil, palm-kernel-oil, poppy-seed oil, etc., are used. Tallow is not well adapted for the purpose. Scraps from soap prepared from cocoanut-oil, etc., may also be utilized. For this purpose, bring, for instance, 200 pounds of water to the boiling point, and dissolve in it 100 pounds of cocoanut-oil soap-scraps, previously reduced to fine shavings by a soap-plane. When all is dissolved, set the stirring apparatus in motion, keeping the contents of the kettle

at a temperature of 99.5° F. A thick, viscid lather is gradually formed, amounting to nearly double the quantity of the original mass. Stirring is continued until the lather is thoroughly homogeneous, which can be readily judged from samples. Shortly before bringing the soap into the frame, it is perfumed with oil of cassia 14 ounces, oil of bergamot 7 ounces, oil of lemon 4 $\frac{1}{4}$  ounces.

The soap being completely cooled in the frame, it is cut into bars, and allowed to dry. The soap may be colored as desired. If only white cocoanut-oil soap-scrap have been worked, it is, as a rule, left white, but colored brown if prepared from colored scrap.

Floating soap may also be prepared by melting a good quality of wax-grain soap or cocoanut-oil soap in water, and converting the mass into a viscid, thick lather by stirring. The porous soap thus prepared can be perfumed and colored as desired, and possesses the property of floating upon water.



## CHAPTER XIX.

VOLATILE OILS AND OTHER MATERIALS USED FOR THE  
PERFUMING OF SOAPS.

THE volatile oils as found in commerce vary very much in quality, and are frequently adulterated. Unfortunately the means for detecting such sophistications are imperfect and difficult to execute, and it is, therefore, the safest plan to purchase these very expensive articles from a source known to be reliable. In the following we give a list of the volatile oils and other materials best known; there are, however, many other oils and substances used in perfuming soaps, but they are of minor importance.

*Oil of bitter almonds* is obtained by submitting bitter-almond cake (left after the expression of the fixed oil of bitter almonds) to distillation with water. It is of a pale, golden-yellow color, colorless when rectified, and has a strong nutty taste and odor. Its specific gravity is 1.043 at 59° F., but varies a little with age.

Oil of bitter almonds is much adulterated with cheaper oils, but chiefly with nitrobenzole, which has an odor very similar to that of genuine oil. *Pure* oil of almond mixed with *sulphuric* acid gives a clear crimson-red color without perceptible decomposition; with an *alcoholic solution of potash* crystals are eliminated. The oil will take up as much as one-third of its weight of *iodine*, and retain the same in solution. *Potassium chromate* does not affect the oil. To detect the presence of nitrobenzole, dissolve 1 part by weight of the suspected oil in 10 parts by weight of alcohol; compound the solution with 1.5 parts by weight of solid caustic potash, and evaporate the mixture to one-third its original volume. Pure bitter almond-oil turns brown, but remains fluid, while, if adulterated with nitrobenzole, a brown resinous substance is formed, which floats in the liquid.

*Oil of bitter almonds* (factitious) known in trade as *nitrobenzole*, *oil of mirbane*, *essence of mirbane*, etc., is now extensively prepared as a substitute for the genuine oil of bitter almonds, and much used for scenting soap, especially the cheaper varieties. It is prepared in the following manner: The apparatus consists of a large glass worm, the upper end of which is divided into two branches, gradually dilating so as to form two funnel-shaped tubes. Concentrated nitric acid is poured into one of the tubes, and benzole into the other. The two substances meet on the point of junction of the two tubes, the rate of flow being regulated by suitable means. Chemical reaction takes place at once, and the new compound is cooled by its passage through the worm which is refrigerated for the purpose. The product is repeatedly washed with water, which completes the operation. Nitrobenzole has a specific gravity of 1.209, boils at  $415.5^{\circ}$  F., and solidifies on cooling to  $37.5^{\circ}$  F.

*Bergamot-oil* is obtained from the rinds of the *Citrus bergamia*, or bergamot-orange. It is of a pale green or yellowish color, becoming, however, darker with age. The specific gravity varies between 0.856 and 0.888 at  $59^{\circ}$  F. and the boiling point between  $365^{\circ}$  and  $397.5^{\circ}$  F. It congeals at  $11^{\circ}$  F. It is one of the most changeable oils and soon acquires an odor of turpentine. It is frequently adulterated with alcohol, being very soluble in it. The alcohol is detected when it is shaken with red sandal-wood, which with a pure oil is not touched, but in alcohol is colored. From the other oils of the family *Aurantiaceæ* it differs by its ready solubility in alcohol: 1 part of oil in  $\frac{1}{2}$  part of alcohol.

*Caraway-oil* is obtained by distillation from the seeds of the common caraway plant, *Carum carui*. The oil is nearly colorless, of a very aromatic odor and acrid taste; it becomes yellow-brownish by age and then shows an acid reaction. It has a specific gravity varying between 0.900 and 0.960 at  $59^{\circ}$  F., and boils at from  $347^{\circ}$  to  $446^{\circ}$  F. It shows but little reaction with iodine and gives an almost clear liquid with alcohol and sulphuric acid.

*Cassia-oil* is obtained by distillation from the buds and barks of *Cinnamomum cassia*, a tree indigenous to China. It is thickly fluid and of a golden-yellow color. Its specific gravity varies

between 1.030 and 1.090. Its fragrance is pleasant and cinnamon-like, but less refined than the genuine cinnamon-oil under which name it is frequently brought into commerce.

*Cinnamon-oil (genuine)* is obtained from the bark of the young branches of *Cinnamomum zeylanicum*, a tree cultivated in extensive plantations in Ceylon. The oil is heavier than water, its specific gravity being 1.005 to 1.050 at 59° F. It is of a pale-yellow to red-brown color, the latter indicating old oil, of an agreeable odor and biting, but pure sweet taste. It remains liquid at —13° F.

*Citron-oil*, from the peel of the fruit of *Citrus medica*, or the citron tree. The rectified oil is colorless, of an agreeable, penetrating odor and acrid taste, and very sensitive to air and light. By exposure to light it turns yellow, and if air be admitted at the same time it is first converted into a fluid, which, on account of its content of ozone, possesses strong bleaching properties. The oil acquires at the same time a disagreeable odor resembling that of oil of turpentine, and is finally converted into a resinous mass. The specific gravity of citron-oil is 0.850 at 59° F., it boils at from 332.6° to 343.4° F., and congeals at —4° F.

*Citronella-oil* is brought into commerce from India and is produced from the leaves of *Adropogon Schoenanthus*, largely cultivated in Ceylon. Its odor resembles that of genuine citron-oil. It is chiefly used for scenting the finer grades of honey and yellow soap.

*Cloves, oil of.*—Every part of the clove-tree, *Caryophyllus aromaticus*, abounds with aromatic oil, but it is most fragrant and plentiful in the unexpanded flower-buds which are the cloves of commerce. They are soaked for some time in salt water and then submitted to distillation. The oil, when fresh, is almost colorless, but on exposure to the air acquires a brownish color and a thickly-fluid consistency. It has the aromatic taste and odor of cloves. Its specific gravity varies between 1.030 and 1.080 at 59° F.; it boils at 482° F. It is frequently adulterated with inferior oils and with carbolic acid. The latter adulteration may be detected by Flückiger's test as follows: Agitate the suspected oil with 50 parts of hot water; decant and slowly evaporate the aqueous portion to a small bulk. Add one



drop of ammonia and a very little chlorinated lime. If carbolic acid be present, a green color changing to a permanent blue is developed.

*Cumin-oil* is obtained by distilling the fresh seeds of the cumin, *Cuminum cyminum*. It is a pale-yellow oil of an aromatic taste and odor. On exposure to air it is rapidly converted into cumic acid. It is lighter than water, its specific gravity being about 0.973 at 59° F.

*Fennel-oil* is derived by distillation from the seeds of the sweet fennel, *Foeniculum dulce*. When pure the oil is colorless, has a hot, pungent taste, and the odor of the plant. Its specific gravity is 0.940 to 0.997 at 59° F.; it boils at from 365° to 374° F. and congeals at from 39° to 64.5° F.

*Geranium-oil, also called ginger-grass oil.*—The oil of commerce which passes under this name, and which was formerly imported from the East Indies, was not obtained from any species of *geranium* or *pelargonium*, but probably from a species of *andropogon*. Properly, however, the term geranium-oil is only applicable to that obtained from some species of *pelargonium*. The genuine oil from the rose-geranium is prepared in large quantities at La Trappe de Staonelli, not far from the Bay of Sidi Ferruch, in Algiers. A finer oil is yielded by the rose-geranium grown in France and Turkey, but it is much dearer. Geranium-oil smells very much like attar of roses, and is for this reason very frequently used for adulterating rose-oil.

*Jasmin, oil of.*—This is obtained from *Jasminum officinale* and *J. grandiflorum*. The fresh blossoms of these two jasmine species are, on account of their small yield of oil, most profitably treated by *enfleurage*. The oil is pale yellow and possesses a pleasant jasmine smell. Good jasmin-oil deposits at 32° F. jasmin stereopts, which crystallize into lustrous scales which melt at 54.5° F. It is lighter than water and is readily soluble in alcohol, ether, and oils.

*Lavender-oil.*—The genuine lavender-oil is distilled from the flowers of *Lavandula vera*; that produced in England possesses the finest fragrance. When freshly prepared it is a colorless liquid, which becomes yellow on standing. It has a hot, camphorous, slightly bitter taste and an odor of lavender. It has an

acid reaction, a specific gravity of 0.875 at 59° F., and begins to boil at 365° F., the temperature quickly rising to 374° F., and the greater portion distilling over between 383° and 419° F. It is frequently adulterated, chiefly with alcohol, but occasionally with oil of bergamot. The pure oil fulminates quickly and violently with iodine, and sulphuric acid turns it reddish-brown, the reaction being accompanied by strong thickening. An admixture of alcohol is readily detected by treatment with a small quantity of tannin. If the latter is not altered, the oil contains no alcohol; if, however, it becomes viscid and sticky, it is adulterated. Adulteration with oil of turpentine is detected by treatment with strong alcohol. For the complete solution of 1 part of oil of lavender 5 parts of 90 per cent. alcohol are required; if, however, the oil contains turpentine, the fluid is turbid.

Another kind of oil of lavender, known as *foreign oil of lavender*, to distinguish it from the English oil, or as *spike oil*, is chiefly obtained by distillation from *Lavandula spica* and *L. stoechas*, or Alpine lavenders. This oil, though very good in itself, cannot compare with fine genuine English oil, and brings only about one-tenth of its price. It has a dark-green color and slight lavender odor; it has an acid reaction, a specific gravity of 0.9081 at 59° F., and begins to boil at from 338° to 347° F.

*Lemons, oil of*, is obtained by various processes from the rinds of lemons. Pure oil of lemons is almost colorless and has the odor of the fruit. Its specific gravity varies between 0.8752 and 0.8785; it boils at 148° F., and is soluble in all proportions in absolute alcohol and glacial acetic acid. It is frequently adulterated with turpentine. This may be detected by slowly heating the oil in a dry test-tube with a small piece of copper butyrate to about 338° F., taking care that the temperature does not exceed 356° F. The copper-salt will dissolve in pure oil of lemon with a green color, while in the presence of oil of turpentine a yellow turbid mixture is obtained; reddish-yellow cuprous oxide being separated. This test is also applicable to oils of bergamot and orange-peels.

*Limes, oil of*, or *limette oil*, is derived from the rind of the lime, *Citrus limetta*. The oil is obtained in the same manner as oil of

lemons, which it somewhat resembles. Its mean specific gravity is 0.8734 at 84° F.

*Marjoram oil* is produced by distilling the flowery tops of the sweet marjoram, *Origanum marjorana*. When freshly prepared, it is yellowish, but becomes brown by age. It has a pungent smell and a hot, peppery and slightly bitter taste. Its specific gravity is 0.911 at 59° F.; when distilled it begins to boil at 365° F., but the temperature rises rapidly to 392° F., and remains constant at between 419° and 428° F., a resinous mass being left in the retort.

*Neroli or orange-flower oil*.—This oil, obtained by distillation of the orange blossoms *Citrus aurantium*, with water, is noted for its fine and fragrant smell and is distinguished over all the other oils of the family *Aurantiaceæ*, in such a manner that a falsification of it seems scarcely possible. It differs, moreover, in its action with nitric acid, by which it acquires a dark red-brown color, while the other oils of the same family of plants are much less colored, some only slightly tinged.

*Nutmeg-oil (volatile)* is obtained by submitting the nutmeg or fruit of *Myristica moschata* to distillation with water. It is colorless or pale-yellow, and has the odor and taste of nutmegs. Its specific gravity is from 0.920 to 0.948 at 59° F. It is soluble in all proportions in glacial acetic acid.

*Orange-peel oil*, also called *Portugal-oil*, is obtained from the peels of the bitter and sweet orange in a manner similar to that of oil of lemons. It is golden-yellow and has a refreshing odor. Its specific gravity varies between 0.836 and 0.890 at 59° F.; it boils at 302° F. It is frequently adulterated with both alcohol and inferior oil of other species of citrus. The latter sophistication may be detected by the different behavior of the oils toward alcohol. Genuine orange-oil dissolves only by repeated shaking with 12 parts of its quantity of alcohol, while inferior citrus oils require only 6 parts. If, therefore, 7 to 9 parts of alcohol are required for the solution of the suspected oil, adulteration is proved.\* With iodine, orange-oil fulminates violently.

*Patchouli-oil* is obtained from the leaves of *Pogostemon patchouli*. It is of a dark-brown color and its specific gravity varies from



0.955 to 0.960. Its odor is the most powerful of any derived from the vegetable kingdom; hence, if mixed in the proportion of volume for volume, it completely covers the smell of all other bodies. It is frequently adulterated to the extent of even 60 per cent. with cheaper oils, mostly those of cedar and cubebs.

*Pimento oil*, or *oil of allspice* from the bruised fruit of the allspice, *Eugenia pimenta*. The oil is pale yellow, becoming reddish-brown by age; it has a very pungent taste and intense odor resembling that of cloves. Its specific gravity varies between 1.021 and 1.037. The pure oil when treated with nitric acid acquires a red color with strong effervescence.

*Rose-oil or attar of roses*.—This precious oil is obtained from various species of roses. The principal commercial source of rose-oil is a circumscribed patch of ancient Thrace or modern Bulgaria, where the variety used for distilling purposes is the so-called Thracian rose, a plant of exceedingly rapid growth. The average quantity of product is estimated by Baur at 0.037 to 0.040 per cent.; another authority says that 3200 pounds of roses give 1 pound of oil.

Pure rose-oil, carefully distilled, is at first colorless, but quickly becomes yellowish; its specific gravity is 0.870 at 72.5° F., its boiling point is 444° F., it solidifies at 52° to 61° F., or still higher, it is soluble in absolute alcohol and in acetic acid. The most usual and reliable tests of the quality of rose-oil are: 1, its odor; 2, its congealing point; and 3, its crystallization. The odor can be judged only after long experience. That of the concentrated oil is intense, penetrating and diffusive, and to most persons unpleasant, the fine and agreeable odor being only brought out by dilution. A good oil should congeal in five minutes at a temperature of 54.5° F., fraudulent additions lowering the congealing point. The crystals of rose-stearoptene are light, feathery, shining plates, and fill the whole liquid.

It is evident that such a valuable oil as rose-oil is very much exposed to adulteration. Indeed, it is said that only adulterated oil reaches the market, the sophistication, chiefly with geranium and ginger-grass oil, taking place at the home of the oil. The general characters of these oils are so similar to those of rose-oil, even the odor bearing a distant resemblance, that detection is very

difficult. Greiner recommends the following as a reliable test: Put one drop of the suspected oil in a dry test-tube and add four drops of concentrated sulphuric acid. A perceptible rise in the temperature takes place, and the mixture must be allowed to stand until it becomes cool. Two grammes of absolute alcohol are then to be added and the mixture well shaken. With pure oil the mixture will be slightly opalescent, and, on heating, will turn yellowish brown, the color remaining on cooling the solution. In the presence of ginger-grass, or geranium-oil, the solution will be turbid and an insoluble precipitate soon forms. Pure rose-oil retains its characteristic odor when subjected to this test, but the mixture with the other oils evolves unpleasant odors.

When fatty oils, such as sesame, almond, etc., are used as adulterants, the usual test, made by placing a drop on white paper, and heating over an alcohol flame, shows their presence in the greasy stain which remains. Pure rose oil is entirely volatile.

The presence of spermaceti, which is usually used for artificially heightening the apparent proportion of stearoptene, is easily recognizable from its liability to settle down in a solid cake, and from its melting at  $122^{\circ}$  F., whereas the stearoptene fuses at  $81.5^{\circ}$  F.

*Rosemary oil* is obtained by distillation from the rosemary, *Rosmarinus officinalis*. It is colorless to pale green, very limpid, and of a more aromatic than sweet odor characteristic of the plant. Its specific gravity is 0.885 to 0.887 at  $59^{\circ}$  F.; it boils at from  $331^{\circ}$  to  $334^{\circ}$  F., and congeals at from  $81^{\circ}$  to  $86^{\circ}$  F. It is frequently adulterated with oil of turpentine; the sophistication can only be detected by the difference of solubility in alcohol. Pure rosemary oil dissolves in an equal volume of 90 per cent. alcohol, while oil adulterated with turpentine requires a far greater quantity for complete solution.

*Sassafras oil*, from the bruised root of the sassafras tree, *Sassafras officinale*. It varies in color from colorless to yellow and red. Its taste is pungent and aromatic, being agreeable to most persons. Its specific gravity varies from 1.070 to 1.090 at  $59^{\circ}$  F. It is frequently adulterated with oil of turpentine, which is,

however, readily detected by the energetic reaction, and by distilling a sample of the oil.

*Thyme oil* is distilled from the flowering herb of the garden thyme, *Thymus vulgaris*. It is pale yellow, of an agreeable aromatic odor, and hot, acrid taste. Its specific gravity varies between 0.877 and 0.875; it boils at from 302° to 455° F.

*Vitiver* or *vetiver oil*, from the rhizome of an Indian grass, *Anatherum muricatum*. It is obtained by distillation either from the fresh root in India, or from the imported dried root. The oil is thickly fluid, of a red-brown color, and has an intense odor very much like that of oil of orris root. Its specific gravity is 0.923 at 59° F; it boils at 515° F.

*Wintergreen-oil* is obtained by distillation from the wintergreen, *Gaultheria procumbens*. The oil found in commerce is generally of a pale-red to deep brown color; one distillation, however, suffices to discolor it completely. It has both a strong and pleasant smell and a warm, aromatic taste. It is the heaviest of all the essential oils, its specific gravity being 1.18 at 59° F. It boils at from 392° to 430° F. It consists of salicylate of methyl and a small amount of terpene, and can be artificially produced from salicylic acid and methyl-sulphuric acid.

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*Peruvian balsam*, according to a report made by Dr. Doret, originates solely from *Myrospermum pereira*. To obtain it, the bark of the tree is beaten in four different places, so that it peels off from the trunk of the tree. A few days after, these places are heated with burning torches, taking the bark away, and placing cloths upon the stripped places, which absorb the oozed-out balsam. These cloths are boiled out in a vessel with water, until they appear entirely free from balsam. The water cooling off, the balsam settles on the bottom. The balsam thus obtained forms a dark-brown, syrupy, opaque liquid, of a very pleasant vanilla or benzoin-like fragrance and aromatic, lasting taste. It is sometimes adulterated by mixing castor-oil with it. To detect this, distil 10 grammes, shaking the distillate, which consists of two layers, in baryta water, taking off the oil-layer floating upon it by means of a pipette, and shaking it with concentrated solu-



tion of bisulphide of soda. In the presence of castor-oil the shaken balsam congeals forthwith into a crystalline mass.

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*Ambergris*.—This is an odorous solid substance found floating on the sea in tropical climates, and in the cæcum of the cachelot or spermaceti whale (*Physeter macrocephalus*). It has been supposed by some to be a morbid secretion of the liver and intestines analogous to biliary calculi; but, according to Mr. Beale, it consists of the mere indurated feces of the animal, perhaps somewhat altered by disease. The color of ambergris is grayish-white and yellow marbled. It has a pleasant musk-like odor, which is heightened by warming, the odor being peculiar, and not easily described or imitated. It does not effervesce with acids; melts at 140° to 150° F. to a yellowish resin-like mass, and at 212° F. flies off as a white vapor. It dissolves easily in absolute alcohol, ether, and also in fat and volatile oils. A factitious ambergris is said to be thus made: Orris powder, spermaceti, gum benzoin, of each 1 pound, asphaltum 3 to 4 ounces, ambergris 6 ounces, grain musk 3 drachms, oil of cloves 1 drachm, oil of rhodium  $\frac{1}{2}$  drachm, liquor ammoniæ 1 fluid-ounce. Beat to a smooth, hard mass with mucilage, and make into lumps whilst soft.

*Civet*.—Under this name is known an animal secretion which originates from *Viverra zibetha*, the Asiatic, and *Viverra civetta*, the African civet. It separates in these animals from particular glands into a sort of pocket which is situated between the anus and the genital organs and opening outside. The wild animal squirts this mass from time to time spontaneously; from the animals kept in captivity it is taken with a spoon. Civet forms a smeary, soft, at first white, after a while brownish mass, becoming in time more consistent. It has a peculiar musk or ambergris-like fragrance and a disagreeable, bitter, irritating taste. It melts when heated, puffs up, takes fire, and burns with a bright flame.

*Musk* is a peculiar concrete substance obtained from *Moschus moschiferus*, an animal bearing a close resemblance to the deer in shape and size, and indigenous to the high plateaus of Asia. The

musk is contained in an oval, hairy, projecting sac, found only in the male, situated between the umbilicus and the prepuce. It is from two to three inches long and from one to two broad. In the vigorous male animal the sac contains sometimes 6 drachms of musk, but in the old seldom more than 2 drachms, and none in the young.

Musk is in grains or lumps concreted together, soft and unctuous to the touch, and of a reddish-brown color resembling that of dried blood. The odor is strong, penetrating, and so diffusive that one part of musk communicates its smell to more than 3000 parts of inodorous powder. The taste is bitter, disagreeable, and somewhat acrid.

Musk is very much adulterated, but very little of the genuine article reaching the market. The Chinese are adepts in this sophistication. Dried blood, on account of its resemblance to musk, is among the most common adulterations, but besides this, sand, iron-filings, hair, the dung of birds, wax, asphaltum, and many other substances are introduced. They are mixed with a small portion of musk, the powerful odor of which is communicated to the entire mass and renders the discovery of the fraud sometimes difficult. The bag containing the musk should have the characters before described as belonging to the natural sac and present no sign of having been opened. Genuine musk burns with a white flame and leaves a white, spongy charcoal; that which burns with difficulty, has a feeble odor and a color either pale or entirely black, feels gritty to the finger, is very moist so as to lose much weight in drying, should be rejected.

*Tincture of ambergris.*—Ambergris (gray) 2 ounces, loaf-sugar 4 ounces, alcohol 8 pints.

*Tincture of civet.*—Civet 2 ounces, orris root (ground) 4 ounces, alcohol 8 pints.

Triturate the civet with the orris root in a mortar, adding the alcohol by degrees.

*Tincture of musk.*—Musk (the best) 2 ounces, sugar 4 ounces, alcohol 8 pints.

These tinctures should be kept in a warm place, occasionally stirring for a month properly to extract the odors, which being of animal origin are difficult of solution.

## CHAPTER XX.

## SOAP ANALYSIS.

IN the analysis of soap we have to consider (1) the content of water, (2) the proportion of fatty acid to alkali, (3) the nature of the alkali and of the fatty acid, as well as of resin, and (4) an intentional admixture of organic and inorganic substances.

*Determination of the content of water.*—In the determination of the content of water special care must be had to obtain a fair average sample. The content of water of the outer portions being less than that of the interior, thin cross-sections must be taken from bars of hard soap and the samples of soft soap from the centre of the barrel. In drying soap containing much water, it frequently happens that it melts when exposed at once to a temperature of  $212^{\circ}$  F. and becomes covered with a film which prevents the escape of the aqueous vapor. To prevent melting together, Loewe recommends first to dry 8 to 10 grammes of the soap, previously reduced to fine shavings at from  $140^{\circ}$  to  $158^{\circ}$  F., and then at from  $212^{\circ}$  to  $221^{\circ}$  F., until the weight is constant. This is best executed upon a large watch-crystal, which for weighing is covered with another one fitting hermetically. For the determination of water, Gladding recommends to weigh a beaker-glass of about 100 cubic centimetres capacity together with a glass-rod. The bottom of the beaker-glass is to be previously covered 1.3 centimetres high with annealed quartz sand. Then introduce into the beaker-glass about 5 grammes of the soap, weigh again, add about 25 cubic centimetres of alcohol, and heat with occasional stirring over a water-bath until the weight is constant. The loss is water. This method is especially recommended for soft soap, which it is difficult to get entirely dry by Loewe's process. The exact determination of water in soap being difficult at the best, as the dried soap readily re-absorbs



moisture, many chemists prefer the indirect manner of determining the content of water, *i. e.*, they determine all the other constituents of the soap and calculate the water from the difference.

Salting out has also been proposed for the determination of the dry content of a soap. The weighed sample is brought into a saturated solution of common salt and heated to boiling. The soap balls together to a nearly anhydrous mass, which is weighed after complete drying. The loss in weight gives the original content of moisture as well as that of nearly all other impurities. This method can, however, lay but little claim to accuracy, as the behavior of soaps from different fats towards salt solution varies very much, and, moreover, the separated soap always retains salt, such being largely the case with soaps from cocoanut-oil and palm-kernel oil.

*Determination of the content of fatty acid.*—Take a sample of 6 to 10 grammes, partially from the interior of the bar and partially from the exterior, in order to obtain one with the average content of moisture. Place the sample in a porcelain dish, and after pouring 20 to 30 times its weight of diluted sulphuric acid (1 part acid to 12 parts water) over it, heat until the clear fatty acid floats on the surface. From oil-soap the fatty acid separates with greater ease than from tallow-soap, but, as it does not completely congeal, it is difficult to remove without loss, from the fluid beneath. This is overcome by the addition of an accurately-weighed quantity (6 to 10 grammes) of thoroughly dried white wax or stearic acid, which is melted together with the fatty acid, the mass after cooling forming a coherent, hard cake, which can be readily lifted from the fluid by means of a spatula. The cake is placed upon a filter and washed with distilled water as long as the latter takes up sulphuric acid, *i. e.*, shows the reaction with barium chloride. The cake of fatty acid is then dried over sulphuric acid, quickest in a rarefied space, until the weight remains constant. Now deduct the weight of the wax or stearic acid added from the total weight of the cake. The remainder, in case soap containing no resin is under examination, represents the hydrate of fatty acid and has to be calculated to anhydrous fatty acid. The content of water of the hydrates of stearic, palmitic, and oleic acids being nearly alike, an allowance of 3.25 per cent.

will be sufficiently accurate for our purposes. Hence 3.25 per cent. is to be deducted from the weight found, the remainder representing the anhydrous fatty acid.

For the determination of *non-saponified fat*, mix the finely pulverized sample of soap with sand, and after drying at 212° F. extract it with petroleum ether. The residue remaining after the evaporation of the petroleum ether may consist of neutral fat or hydrocarbons. The presence of the latter might be due to the fats used in the preparation of the soap containing mineral oil, or hydrocarbons having been mixed with the soap. By testing the residue as to whether it is saponifiable or not, its nature is readily established. The statement which is frequently made that the presence of free fat is indicated by the unctuous feel of the soap cannot be relied upon, since soap in the preparation of which cotton-seed oil has been used, shows also an unctuous feel.

The question *what kinds of fats have been used in the preparation of the soap* is very difficult and frequently impossible to answer by the chemist, the only guides being the determination of the melting point, equivalent of saponification and iodine number of the separated fatty acids. The determination of the fatty acid with the assistance of wax or stearic acid, being not available for this purpose, another sample of the soap has to be decomposed by means of acid and the melting point, equivalent of saponification and iodine number of the resulting fatty acids determined by the methods previously given.\*

*Determination of resin.*—The presence of resin is, as a rule, readily recognized by the color and odor of the soap, but the determination of the quantity of it is more difficult. According to Gladding, 0.5 gramme of the fatty acid obtained by decomposing the soap with acid is shaken in a cylinder, graduated to 100 cubic centimetres with 20 cubic centimetres of 95 per cent. alcohol until all is dissolved. The solution is then compounded with one drop of phenol-phthaleïn solution and after adding concentrated potash-lye until the alkaline reaction takes place, heated in a water-bath. After cooling the cylinder is filled up to the 100 cubic centimetre mark with ether, and, after shaking, 1 gramme of dried

\* See pp. 85, 90, and 91.

and pulverized nitrate of silver is added, and the whole shaken until the precipitate balls together in a manner similar to chloride of silver. After allowing the precipitate consisting of silver salts of fatty acids to subside, 50 to 70 cubic centimetres of the supernatant clear fluid are brought by means of a pipette into a cylinder graduated into 100 cubic centimetres and shaken with a very small quantity of nitrate of silver, in order to be sure that everything has been precipitated. If a precipitate is formed, it is best to commence the test anew. If there be no precipitate, shake with 20 cubic centimetres of dilute hydrochloric acid (1 part acid, 2 parts water) and after allowing the chloride of silver to subside read off the height of the ether-layer. Then bring an aliquot part of the layer of ether into a platinum dish, evaporate to dryness, dry at  $212^{\circ}$  F., and weigh the residue which is brought into the calculation as resin. According to Gladding, a small correction of 0.002359 gramme for every 10 cubic centimetres of the solution can be made for the small quantity of oleic acid which has passed into solution.

*Determination of alkalies.*—The alkali contained in soap is either potash or soda, or a mixture of both. To determine the kind of alkali, separate the fatty acids from a solution of soap and test with alcoholic solution of chloride of platinum which, in the presence of potassium, gives a yellow precipitate. The quantitative determination of the alkalies, if only one be present, is effected by the alkalimetric method. Decompose the soap with an excess of normal acid, and after allowing the fatty acids to separate, determine the quantity of acid added in excess by retitrating with caustic alkali. The difference gives the quantity of acid used for the neutralization of the alkali of the soap and from it the quantity of the alkali itself is found. If the decomposition of the soap is not readily effected by the acid, add some spirit of wine, which dissolves the soap and facilitates its decomposition. Heating or boiling should by all means be omitted.

If both potassium and sodium are present, the total quantity of alkali is alkalimetrically determined in one sample, while a second sample is decomposed with hydrochloric acid and the potassium in the solution determined with chloride of platinum.



The sodium is then calculated from the potassium found and the total quantity of alkali.

The following method may also be used for determining the alkalies: The quantity of soap weighed off is brought into a platinum crucible and after carefully incinerating the organic substance, the ash is treated by the alkalimetric method. For ordinary purposes it is sufficient to give the entire content of alkali calculated from the titration as sodium oxide ( $\text{Na}_2\text{O}$ ) for hard soaps and as potassium oxide ( $\text{K}_2\text{O}$ ) for soft soaps.

*Whether a soap contains free alkali, i. e., caustic alkali or carbonate* is recognized by the addition of a small quantity of phenolphthaleïn to an alcoholic solution of the soap which produces a red coloration; further by dropping upon the freshly-cut surface of the soap a small quantity of solution of mercuric chloride, which produces a yellow coloration, or a small quantity of mercurous nitrate, which gives a black coloration. The test with mercuric chloride is, however, not always reliable.

For the determination of *non-saponified alkali*, boil the weighed-off sample with distilled water, and introduce common salt in small portions into the solution. The soap coagulates and separates out, while the non-fixed alkali remains in the salt solution. Now continue the addition of common salt until the last portions added do not dissolve. Then bring the salt solution into a beaker glass, and, after adding the wash-water used in freeing the soap from adhering saturated salt solution, determine the quantity of alkali by the alkalimetric method, having previously ascertained its presence with curcuma paper or litmus paper.

*Determination of glycerin.*—Dissolve about 25 grammes of the soap to be examined in hot water, and compound the solution with dilute sulphuric acid until acid reaction takes place. Then melt the fatty acids together with wax, and, after cooling, lift off the cake of fat, and evaporate the fluid previously exactly neutralized with sodium carbonate to dryness in a water-bath. Treat the residue, consisting of sodium sulphate and glycerin, with alcohol, whereby the sodium sulphate remains undissolved behind. Then evaporate the alcoholic solution; again treat the residue with alcohol, and evaporate the filtered solution in a platinum dish in a water-bath. Great accuracy cannot be claimed for this

method on account of the volatility of the glycerin. For a more accurate determination of the glycerin, dissolve, according to the suspected content of glycerin, 1 to 10 grammes of the soap in water, or, if organic substances insoluble in water be present, in methyl alcohol. Filter the solution, and after evaporating the methyl alcohol, if used, separate the fatty acid, and then proceed with the acid filtrate in the same manner as for the determination of glycerin in fats.

*Determination of alcohol.*—Transparent soaps being, at the present time, prepared with alcohol as well as without, it may sometimes be important to know whether a soap contains alcohol. For its determination, mix, according to Valenta, 50 to 60 grammes of the soap to be examined with pumice stone, and distil in a paraffin-bath, first at  $230^{\circ}$  F. and then at  $248^{\circ}$  F. With the distillate the iodoform test is made, which is executed, according to Hager, as follows: The fluid to be tested is compounded with 5 to 6 cubic centimetres of a 10 per cent. potash solution, and after heating to from  $104^{\circ}$  to  $122^{\circ}$  F., 16 to 20 per cent. potassium iodide solution saturated with iodine is added until the fluid assumes a yellow-brownish color. If the color does not appear on shaking, add by means of a glass-rod just sufficient potash lye entirely to decolorize the fluid. Immediately, or after standing a short time, yellow crystals of iodoform are separated out, which, when viewed under the microscope, present a star-like appearance, or one of hexagonal tablets.

*Determination of volatile oils.*—The separation of volatile oils used for perfuming soaps can, according to Barfoed, be effected by two methods. Extract the soap at an ordinary temperature with ether, filter through a filter moistened with ether, and rinse off with the same liquid. Shake the solution with water to remove any soap which may have passed into it, and then evaporate; or dissolve the soap in water, compound the solution with a small quantity of sulphuric acid to prevent strong foaming, and distil off the volatile oils. From the distillate the oil is collected by shaking with ether.

*Determination of filling agents.*—The filling agents are partially salts soluble in water, especially the chlorides and sulphates of potassium and sodium, the alkaline carbonates and water-glass,

partially mineral substances soluble in water, such as talc, heavy spar, infusorial earth, etc., and partially organic substances, such as potato flour. For the determination of admixtures, dissolve the soap, previously cut into fine shavings, in 8 to 10 times its quantity of 90 per cent. alcohol by moderate heating in the water-bath. Then filter the solution, and, after washing the residue with alcohol, and drying at  $212^{\circ}$  F., weigh it. Transparent soaps prepared with the assistance of spirit of wine alone are soluble in alcohol without leaving a residue.

*For determining the portion of the residue soluble in water*, extract it with cold water, and after reducing it to a determined volume, take portions of it for determining the sulphuric acid, chlorine, silicic acid, and the alkalies. In the absence of water-glass, the carbonic acid fixed on alkalies can only be determined by titration, direct determination of the carbonic acid being otherwise preferable. A content of water-glass can also be determined by dissolving the soap in water, and compounding the solution with an acid. The fatty acids float on the top, while the silicic acid falls to the bottom, or remains suspended in the fluid. It is collected upon a filter, washed, dried, heated, and weighed.

Soap containing water-glass yields free alkali to spirit of wine, while the silicic acid remains behind as a jelly.

By boiling the portion insoluble in alcohol with water, a thickish solution is formed in the presence of *starch*, which acquires a blue coloration on adding a few drops of tincture of iodine. To test for *glue*, extract the portion of the soap insoluble in alcohol with hot water. The solution gelatinizes on cooling, and yields a precipitate on compounding it with solution of tannic acid. *Mineral oils* and *mineral fats* can be readily determined by being non-saponifiable.

For the determination of *talc*, *heavy spar*, *infusorial earth*, etc., heat the residue insoluble in water in order to destroy the organic substances, and then test it as to its constituents.

*Prof. Albert R. Leeds's scheme for the analysis of soap.*—(1) *Water*: Weigh out about 5 grammes in very fine, small shavings upon a dried, weighted, plated filter. Dry at  $230^{\circ}$  F. until the weight is constant. The loss is water. (2) *Uncombined fat*:



Transfer the filter containing the dried soap to the funnel connected with the return-cooler, such as is used in the determination of the albumenoids in milk, and connect with the funnel a small tared flask containing 50 cubic centimetres of petroleum ether. After complete extraction distil off the ether, and the residue in the flask, dried at  $230^{\circ}$  F., will be the uncombined fat.

(3) *Free alkali*, (4) *Glycerin*: Allowing the funnel, with the soap freed from moisture and from fat, to remain on the return-cooler, attach to it a flask containing 75 cubic centimetres of 95 per cent. alcohol, and extract. To the alcoholic solution add a few drops of phenol-phthaleïn; if free alkali be present, neutralize with normal sulphuric acid and calculate the amount of uncombined soda. After neutralization add a large excess of water and boil off the alcohol. To the aqueous solution add a large excess of normal sulphuric acid. Boil, cool, and decant through a small filter, wash with hot water, and decant, after cooling, through the filter until litmus-paper is no longer reddened by the washings. The filtrate consists of the combined soda and glycerin, the residue of the fatty acids, and resin. Neutralize the filtrate with normal soda solution and calculate the amount of combined soda as  $\text{Na}_2\text{O}$ . Evaporate to dryness and extract the glycerin with absolute alcohol. Transfer the alcoholic solution to a tared flask, distil off the alcohol, dry at  $212^{\circ}$  F., and weigh the residue as glycerin.

(5) *Fatty acids and resin*: Dissolve the small amount of the fatty acids and resin that may be on the filter, through which the decantation was effected, with a little petroleum ether; add the solution to the larger bulk in the beaker, evaporate off the ether, dry at  $212^{\circ}$  F., and weigh the combined fatty acids. Multiply this result, after subtracting the amount of the resin, by 0.97, and the product is the fatty anhydrides.

(6) *Resin*: The resin was separated from the fatty acids according to the methods proposed by Gladding. About 0.5 gramme of the mixture of the fatty acids and resin is dissolved in 20 cubic centimetres of strong alcohol, and with phenol-phthaleïn as an indicator, soda is run in to a slight supersaturation. The alcoholic solution, after boiling for ten minutes to insure complete saponification, is mixed with ether in a graduated cylinder till the volume is 100 cubic centimetres. To the

*Prof. Leeds's scheme for soap analysis.*

Weigh out 5 grammes. Dry at 212° F. Loss corresponds to water.

Treat with petroleum ether.

Residue in soap and mineral constituents. Treat with alcohol.

Residue.— $\text{Na}_2\text{CO}_3$ ,  $\text{NaCl}$ ,  $\text{Na}_2\text{SO}_4$ , sodium silicate, starch, and insoluble residue. Wash with 60 c. c. water.

Filtrate.— $\text{Na}_2\text{CO}_3$ ,  $\text{NaCl}$ ,  $\text{Na}_2\text{SO}_4$ , and sodium silicate. Divide into four equal parts.

Residue.—Starch and insoluble residue. Dry the filter and weigh. The weight is the starch and insoluble residue. Starch.—Convert the starch into  $\text{C}_6\text{H}_{12}\text{O}_6$ . Titrate with Fehling's solution. Subtract the weight of starch found, and the difference is the insoluble mineral constituents.

Add a large excess of water and boil off the alcohol. Decompose with excess of normal  $\text{H}_2\text{SO}_4$ . Boil, filter, and wash.

Residue.—Fatty acids and resin. Dry at 230° F. and weigh. Dissolve an aliquot part in 20 c. c. strong alcohol, and, using phenol-phthalin as an indicator, saponify with soda in slight excess. Boil, cool, and add ether to 100 c. c. Decompose with  $\text{AgNO}_3$  by adding in fine powder, and shake well for ten minutes. Allow to settle.

Solution.—Resinate of silver. Filter 50 c. c. from the total 100 c. c. Decompose with 20 c. c.  $\text{HCl}$  (1 : 2). Allow the  $\text{AgCl}$  to settle, and evaporate an aliquot part of the ethereal solution in a tared dish. Dry at 230° F. and weigh. After applying correction for oleic acid, the weight corresponds to the resin. This weight subtracted from the combined weight of fatty acid and resin gives the fatty acids.

Extract is uncombined fat. Dry at 230° F. and weigh.

$\text{H}_2\text{SO}_4$  used corresponds to free alkali. Calculate as  $\text{NaHO}$ .

Filtrate.—Combined soda and glycerin. Titrate with normal soda solution.

After titration evaporate to dryness on the water-bath. Treat with absolute alcohol. Evaporate the alcoholic solution to dryness in a tared dish and weigh as  $\text{Na}_2\text{O}$ , glycerin.

Precipitate is stearate, palmitate, and oleate of silver.

alcoholic and ethereal solution 1 gramme of very finely powdered  $\text{AgNO}_3$  is added, and the contents of the cylinder are shaken thoroughly for ten or fifteen minutes. After the precipitate has settled, 50 cubic centimetres are measured off, and if necessary filtered into a second graduated cylinder. A little more  $\text{AgNO}_3$  is added to see if the precipitation is complete, and then 20 cubic centimetres of dilute hydrochloric acid (1 : 2) to decompose the silver resinate. An aliquot part of the ethereal solution in the cylinder is evaporated in a tared dish, and weighed as resin, deducting a small correction (for 10 cubic centimetres deduct 0.00235 gramme) for oleic acid. The amount of resin subtracted from the combined weight of fatty acids and resins, as found before, gives the fatty acids. (7) *Sodium carbonate*, (8) *Sodium chloride*, (9) *Sodium sulphate*, (10) *Sodium silicate*, (11) *Insoluble residue*: The filter in the funnel connected with the return-cooler, after treatment with alcohol, contains the mineral constituents of the soap. The contents of the filter are washed with cold water till the washings amount to 60 cubic centimetres. The filter is then dried and weighed. The weight gives the insoluble residue and starch. The starch is converted into glucose with dilute acid, and titrated with Fehling's solution. The weight of starch found, subtracted from the total weight of insoluble residue and starch, gives the insoluble mineral constituents. The aqueous solution of 60 cubic centimetres, just mentioned, is divided into four equal parts, in one of which is determined the carbonate of soda by titration, and in the other parts the chloride, the sulphate, and the silicate respectively by any convenient method.



## PART II.

### THE MANUFACTURE OF CANDLES.

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#### CHAPTER I.

##### INTRODUCTION.

*Historical notice.*—Of all the means of artificial illumination candles are perhaps the most convenient, and the materials of which they can be made are generally easily obtained and many of them cheap in cost. The manufacture and introduction of candles amongst the domestic conveniences of life, were in a manner no less productive of refinement in the taste and habits of communities than were those of soap and glass. It is difficult to trace from history the first introduction of the candle. Illumination by its means was not known to the ancient Egyptians and Jews. Though in Holy Writ many references are made to candles and candlesticks, upon further consulting the same sacred record it will be observed that these terms were used either in a metaphorical sense, or otherwise the translators have been at fault in rendering the meaning of a word in the Oriental tongue by one which does not express the same in modern languages. That the candlesticks spoken of were intended to support lamps, not candles, is plain from the instructions Moses received from the Almighty for making the golden candlestick. “*And thou shalt make the seven lamps thereof, and they shall light the lamps thereof, that they may give light over against it.*”\* Further testimony showing that olive-oil was employed for those lamps may be found in the Book of Leviticus, from which, as well as from the foregoing, it is plain that candles were not in use among the ancient

\* Exodus XXV., 37, 38.

Jews. The Romans were equally ignorant of the candle, and although Pliny and other writers mention it, yet the only information to be gathered from them is that their candles consisted of strings of flax, saturated and covered over with pitch or wax, probably not unlike those formerly used by laborers for working at night. Pliny also records the use of the pith of reeds saturated with fat as a night-light, which was placed alongside a corpse as long as it remained in the house. He also mentions strips of papyrus and reeds, steeped in pitch and then coated with wax. According to some statements, the manufacture of candles is contemporary with the persecutions of the early Christians by the Roman Emperors, particularly when the former took refuge in the catacombs from the rage of their oppressors. This assertion is rendered more probable from the fact that it is customary since that period to burn candles in all church ceremonies of the Roman church, and Apuleius, near the end of the second century, makes a distinction between wax and tallow-candles (*cerei* and *sebaei*).

Beckmann, in his "History of Inventions," relates that the Emperor Constantine, who ruled about the beginning of the fourth century, caused the whole city of Constantinople to be illuminated with lamps and wax candles one Christmas eve, and that the night had been lighter than the brightest day.

In the middle ages the use of wax candles and wax torches for church and household purposes was well known, and Fosbrook mentions that the wicks were made of twisted tow and the candles were cast in moulds and varied from a very small size to fifty pounds in weight. At first they were, very likely, only used in the houses of the rich, as wax was scarce and commanded a high price, which is best proved by the fact that Philip the Bold, Duke of Burgundy, in 1361, offered to present St. Anthony, of Vienna, with his weight in wax for the cure of his sick son.

Before the invention of clocks the consumption of a wax candle of determined length and thickness served frequently for the approximate determination of time.

On account of the rites and ceremonies of the Catholic Church, the consumption of wax candles became enormous, but decreased somewhat by the spread of Protestantism, to be, however, abun-

dantly made up by the luxury of courts, especially in the eighteenth century. Before the Reformation 35,730 pounds of wax candles were, for instance, annually consumed in the Cathedral at Wittenberg, and at a single court entertainment in Dresden, 1770, 14,000 wax candles (about 675 pounds) were burned. An idea of the enormous consumption of wax candles at the Berlin Court during the reign of Frederick William II. may be had from the fact that an annual defalcation of about \$4500 in their purchase remained undiscovered for many years.

Tallow candles were already known in the twelfth century, but came into general use as a cheaper illuminating agent only in the fifteenth century. Candles remained, however, too scarce and high-priced for use among the masses, and they do not seem to have been in general use until the middle of the last century, nor was there any important improvement in the art from the first crude methods of dipping, except that they were about that period moulded in metal moulds. Indeed, up to our own century very little improvement can be traced, nor did candles receive their due importance or approach their present perfection until the discovery of the elements of the fatty bodies and their decomposition into the fatty acids, stearic and palmitic.

When in 1830 paraffin was discovered by Reichenbach, candles were still further improved by the addition of this valuable substance to stearic acid to prevent crystallization, or the stearic acid was combined with the paraffin to improve them and prevent their softening and bending in a warm atmosphere.

Candles of wax or tallow were first made by dipping, though the former were sometimes made by drawing and rolling, a mode still in vogue. The moulding of candles is of quite a recent date, for though they were moulded after a fashion over a hundred years ago, they were not made systematically until 1820. At the present time, owing to the many improvements in the materials and in the appliances for moulding, the manufacture of candles may be said to have reached perfection.

*Evolution of light by the candle.*—The functions of the candle as an illuminating material are by no means as simple as they would appear at the first glance. Frederick Knapp says: "The candle may be considered as a real microcosm of illumination, in



which all the individual functions regulate each other. The curious manner in which the separate functions mingle in a candle and pass one into the other, in carrying out the main object, as well as the force of long habit, render the very same apparatus (as far as the principle is concerned), performing the same operations, less remarkable to the casual observer than gas illumination, which, being carried out on so extensive a scale, and at a vast expense, excites more general astonishment."

A very important part of the candle is the wick, it being the intermediary link between the fatty substance and the flame. It consists of a bundle of fibres and dips with the lower end into the fluid illuminating substance and with the upper into the flame.

The original, and not yet obsolete material employed for wicks, was the common soft rush *Juncus conglomeratus*, to be found in moist pastures, and by the sides of streams and ditches. Fine cotton-yarn is, however, the principal material used at the present time. Turkish-cotton rovings are said to be the best, but of the cotton employed for this purpose there is certainly a great deal more grown in the United States than in Asia Minor.

Several processes must actually be distinguished in a burning candle, each of which must keep within correct bounds, in order to produce, by their organic mingling into each other, a steady, bright, and faultless flame. These processes may be divided into two physical and two chemical ones:—

1. The melting of the illuminating material.
2. The absorption of the melted material by the wick.
3. The decomposition of the melted material into gaseous and vaporous products.
4. The combustion under the most favorable circumstances for the development of light.

The burning candle must, therefore, accomplish by itself that with which a lamp is provided with from the start—the fluid illuminating substance, which in a lamp can be regulated by the manner of construction, the supply of air, and the conveyance of oil to the wick.

In a burning candle the fat below the flame is melted into the form of a hollow cup by the heat constantly radiating in all directions. A reservoir is thus formed in which that which is

melted by the heat uninterruptedly collects, and from whence it is at the same time conveyed to the flame for its nourishment. If, now, on the one hand, the illuminating material melts at a greater ratio than corresponds to its capability of decomposing and the ability of absorption of the wick, the candle will gutter, and if, on the other, all the gasified illuminating material is not consumed, the flame will soot and languish by the deposited soot obstructing the capillary channels of the wick. Such deposit of carbon frequently occurs, as is well known, especially in the flame of a tallow candle.

One of the principal conditions for the regular process of illumination is that the wick should stand in the centre of the candle, and, besides capillarity, must possess another property quite as essential. The flame is always produced at that part of the free wick which is in the middle between its point of most active capillary action and the point where the flow of melted fat is excessive, therefore always at the same distance from the bottom of the above-mentioned hollow cup. The wick, therefore, if the candle is to regulate itself, must shorten as the candle diminishes, *i. e.*, it must be prepared from a substance which is combustible in the heat of the flame. The wick-yarn of commerce consists, as previously stated, of cotton rovings. The wick of tallow-candles is much thicker and more abundantly saturated with fat than that of stearin candle, because tallow melts at from  $98.5^{\circ}$  to  $104^{\circ}$  F., while wax and stearic acid, as found in commerce, melt at somewhat above  $140^{\circ}$  F. A flame developing a determined degree of heat will, therefore, melt in a given time and, at a determined distance from the candle, more tallow than stearic acid. This is, however, not the case with our ordinary candles, a tallow-candle not being consumed quicker than a wax or stearin-candle of the same thickness.

From the position of the wick in the axis of the candle, the requisite quantity of air, being used up by the flame itself, does not reach the wick, and completely consume it. The fibres of cotton are, therefore, charred, and they remain so until some part projects beyond the flame, and, coming into contact with the air, is consumed. If this happens, as it generally does, too late, then soot collects, in consequence of the interruption of the flame, as a

spongy stuff on top, darkens the flame, and falls eventually, if not removed with the snuffers, into the above-mentioned hollow cup, where it causes further interruption of the process. The thin, and especially the plaited, wicks of wax and stearin-candles have the property of bending as soon as a part projects beyond the flame, and being thus constantly reduced to ashes, snuffing is not required. By this the symmetry of the flame is, of course, partially destroyed, as it follows the inclination of the wick, and consequently melts too much fat on the one side, which gutters over the melted edge of the cup.

Generally speaking, no candle exhibits an equilibrium of the above-mentioned points, whatever be the material of which it is composed—tallow-candles generally much less so than others—for, independent of the fusibility of the material, it is influenced by the relation which the mass of fat, *i. e.*, the thickness of the candle, bears to the size of the wick, a relation which has been established approximately by long experience. The usual dimensions of a candle are, therefore, not fixed arbitrarily or by chance, but are absolutely necessary to a well-regulated process of combustion.

The principal task of the illuminating material is to furnish gases for the production of a brightly burning flame. Every fluid or solid body which vaporizes or gasifies under decomposition at a temperature lower than that required for combustion can, of course, only burn as gas, and the phenomenon of light thereby observed is called "the flame." It acquires its well-known form by the pressure of the air surrounding it, which, as a gaseous body rendered lighter by the heat, is displaced, and caused to ascend. The wick directly contributes nothing to the development of light, and may consist of capillary mineral substances (asbestos), fine glass tubes, etc., though the latter, on account of conducting more heat, would melt the fat around the wick too much.

The flame, at first small and of little illuminating power, increases to within a certain limit with the progressive decomposition of the fat. Moreover, the decomposition of the melted fat does not take place suddenly, but gradually, *i. e.*, in the degree as, in ascending in the wick, it reaches the hotter points of the flame.



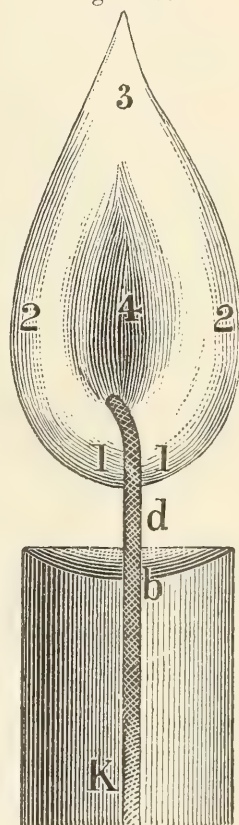
It is first decomposed into its immediate constituents—stearic, palmitic, and oleic acids, and glycerin—and these into products of decomposition of a higher order—margaron, acrolein—and the latter finally into permanent gaseous hydrocarbons, *i. e.*, such as contain no more condensable bodies, marsh-gas, etc., as well as varying quantities of hydrogen, carbonic acid, etc., and vaporous, incompletely gasified substances.

The first and most important elucidation in regard to the nature of the flame and of illuminating itself, we owe to the researches of Sir Humphrey Davy. In modern times many chemists have devoted much time to the study of the flame, the labors of Hilgard, Landolt, Deville, Knapp, etc., having much contributed to our knowledge.

The evolution of the burning gases, as well as their combustion, and the shape of the flame can be nicely observed in a lighted candle, *K*, Fig. 100. The melted illuminating material *b* is absorbed by the wick *d*, and converted into gas, which, flowing out from the point of the wick, surrounds the latter in the shape of a cone. For distinct parts can be recognized in this cone of gas the lower part (1), which burns with a pale-blue flame; the veil (2) of very weak illuminating power. It consists of burning carburetted hydrogen, and surrounds the actual luminous cone of flame, (3) which also consists of burning carburetted hydrogen, in which are diffused glowing hydrocarbons. The dark central nucleus (4) consists of gases not in a state of combustion.

The air surrounding the current of gas arising from the candle can only penetrate to a certain depth, as seen by the limits of the luminous cone of flame. The conical shape of the flame is explained by the interior masses of gas not in a state of combustion pushing upwards in columns, becoming gradually thinner, similar to the parts of a spy-glass, and reach-

Fig. 100.



ing combustion by degrees. That the temperature of the flame is highest on the edges of the luminous cone, and decreases towards the nucleus, is readily proved by holding a match cross-wise in the flame, when the portion immediately on the edge of the cone will be strongly charred, while the intermediate portions of the wood remain white.

The consumption of oxygen caused by the illuminating materials under medium atmospheric pressure is, according to the materials used, as follows :—

1 kilogr. of tallow	consumes the oxygen from 16.35 cubic metres of air.						
1    “    wax	“	“	“	10.41	“	“	
1    “    rape-oil	“	“	“	12.21	“	“	
1    “    illuminating gas	“	“	“	13.62	“	“	

The amount of carbonic acid generated does not exceed that produced by the process of breathing and is on an average about 0.4 cubic metre per hour.

If by blowing out the flame of a tallow candle, the temperature is so far reduced that the fat contained in the point of the wick is decomposed but a combustion of the products of decomposition cannot take place, the product of decomposition of glycerin, known as acrolein, is developed.

The test of the illuminating value of a fat, oil, or gas serving for illumination (photometry) can only be a comparative one, *i. e.*, it can only be determined how much more or less effect is produced by one than by the other. The quality of an illuminating agent depends (1) on the intensity of the light it produces as compared with another material, and (2) on the consumption of material in a determined time to produce the same intensity of light.

The performance of an illuminating material increases therefore with the decrease in the consumption of material, and consequently is equal to the intensity of the light divided by the consumption of material. The quantity of solid or fluid illuminating material consumed is ascertained by weighing before the experiment and reweighing after burning for a certain measured duration. With illuminating gas the volume consumed in a certain time is measured.

In photometric experiments it must be carefully observed that

the intensity of the light of a flame does not alone depend on the material and the consumption, but also on the apparatus in which it is burnt. The various shapes of burners for gas are, for instance, not at all alike in their performances; it is further not indifferent in what kind of lamp the oil is burned, and it depends even on the thickness of the wick and the diameter of the candle, whether the performance of a candle material is more or less good. To test, therefore, the value of a material in every respect, the experiments should be executed with various forms of apparatus and material.

The intensity of the light is determined by instruments known as photometers. As far as used for technical purposes they are based upon the physical law, *that the intensity of the illumination of a plane decreases in the ratio in which the square of the distance between it and the source of light increases.*

Two unequal intensities of light can only be made comparable by changing the distances of a screen catching the light until the effects of both are alike and then measuring the distances.

A photometer much in use at the present time is based upon a principle first applied by Bunsen. Upon one side of a long horizontal rod stands the normal light (a paraffin candle, six of which = 1.1 pound) which is used as a unit and the flame of which, for every experiment, is brought to a determined length by trimming the wick, etc. Upon the other side is the candle or other source of light to be examined, and between both is a movable screen of thin paper rendered semi-transparent by being saturated with a solution of spermaceti in oil of turpentine, with the exception of a central spot which is allowed to remain opaque. In using the apparatus (in a dark room) the standard light is placed behind the spot and the light to be examined in front. When the two surfaces are equally illuminated, the opaque spot disappears and the whole surface of the disk is perfectly homogeneous in appearance.

To save the calculation of the intensity of the light of the illuminating material to be examined from the distance of the screen, the intensity of light corresponding to every position of the screen is marked upon the rod.

The simplest and most readily constructed photometer is that





## CHAPTER II.

## MATERIALS FOR CANDLES.

IN addition to tallow, palm-oil, Chinese tallow, and the various fats and oils described in Chapter V., and of stearic acid, the manufacture of which by the various processes is described and illustrated in Chapter II., there are several other classes of hydrocarbons which are largely used by the candlemaker. Perhaps the material next in importance to stearic acid is

*Paraffin*, a product of the distillation of several organic bodies under high temperatures. The discovery of this body fairly belongs to Karl Reichenbach, who gave to the strange compound its present name, from *parum* and *affinis*, because it appeared to him to be wholly destitute of chemical affinities. His discovery and its peculiar behavior in this respect were published in the year 1830. The following year its presence in the petroleum of Rangoon was noticed by Christison of Edinburgh. He called it *petroline*, but hearing subsequently of Reichenbach's discovery, he was satisfied of the identity of the two substances and withdrew the name given by himself. In 1833, 1834, and 1835, we have records of the separate discoveries by the three chemists Laurent, Gregory, and Viobel, the former showing that the oils distilled from shale contained paraffin. The credit of founding the commercial industry based upon the manufacture of paraffin and its allied products belongs to no one person more properly than to Seligue, of France, and to his genius and indefatigable industry belong the many improvements made in the manufacture of oil from bituminous shale. From Prof. F. H. Storer's remarks on the discoveries of Seligue in connection with the paraffin industry we make the following extract, which succinctly details the various processes secured by him :—

“On the 27th of March, 1839, Seligue specifies certain addi-

tions and improvements to a former patent. In alluding to the use of his oils in the treatment of cutaneous diseases, he speaks of the three large establishments for the distillation of bituminous shale, which he has erected in the department of *Sône et Loire*, and mentions the fact that the crude oil is furnished at the rate of ten centimes per pound. The clearest of all of *Seligue's* specifications, however, is that of the patent granted to him March 9, 1845, for the bituminous shales and sandstones. After describing the various forms and apparatus used in distilling, into one of which superheated steam was introduced, he enumerates the products of distillation as follows: I. A white, almost odorless, very limpid mineral-oil, somewhat soluble in alcohol, which may be used as a solvent or for the purpose of illumination in suitable lamps. II. A sparingly soluble mineral-oil of 0.84 to 0.87 specific gravity, of a light-lemon color, perfectly limpid, almost odorless, never becoming rancid, and susceptible of being burned in ordinary lamps having an elevated reservoir, with double current of air, a slight modification of the chimney and burner being alone necessary. This oil can be mixed with the animal and vegetable oils. Oils thus prepared do not readily become rancid, nor do they congeal easily when subjected to cold. III. A fat mineral-oil, liquid at the same temperature as olive-oil. This oil contains a little paraffin and is peculiarly adapted for lubricating machinery, having an advantage over olive and other vegetable oils, or neat's-foot oil, in that it preserves its unctuousness when in contact with metals and does not dry up. IV. From the oils I., II., and III., I extract a red coloring matter, which can be used in various arts. V. White crystalline paraffin, which needs but little treatment in order to be fit for making candles. This substance does not occur in very large proportion in the crude oil, and the proportion varies according to the different mineral substances upon which I operate. There is but little of it in petroleum and in the oil obtained from bituminous limestone. I often leave a great part of the paraffin in the fat oil and in the grease in order that these may be of superior quality. VI. Grease. This grease is superior to that of animals for lubricating machinery and for many other purposes, since it does not become rancid and remains unctuous when



in contact with metals. VII. Perfectly black pitch, very drying, suitable for preserving metals, wood, etc. VIII. An alkaline soap obtained by treating the oils with alkalies. IX. Sulphate of ammonia. X. Manure, prepared by mixing the ammoniacal liquor, or the blood of animals, with crushed fixed residue (coke) of the shale. XI. Sulphate of alumina from the residue of the shales."

To Dr. James Young belongs the honor of creating the paraffin industry in Scotland. In 1850 he took out his celebrated patent for the distillation of coal minerals at a temperature not exceeding 600° F.

According to G. T. Beilby the oil-shale belt extends right across Scotland, from Ayrshire and Renfrewshire on the west to Midlothian and Fifeshire on the east. The minerals worked in the west are among the upper series corresponding closely to cannels or coals; those worked at the centre of the belt between Shotts and West Calder are intermediate, while those in Midlothian and Fife are lowest. It is curious that the western minerals yield the largest quantity of oil per ton, but of the poorest quality, while the eastern minerals give the smallest yield per ton, but the oil is of the finest quality; and in this respect the central district in the belt is intermediate between east and west, the quantity and quality of the oil being an average. Oil shales are mined and brought to the surface in much the same way as coal.

The crude oil obtained from the shales is subjected to fractional distillation, and the last fraction is very heavy, containing 30 per cent. of paraffin. The hard scale separates from the oil by crystallization in ordinary weather, and the residual oil is then artificially cooled and yields a further quantity of soft scale. The crude, solid product thus formed is known as "paraffin scales," and is of a somewhat variable composition. The impurities amount, on an average, to 20 per cent. of the weight, and consist of blue oil, greasy hydrocarbons of low fusing point, solid refuse, and water. For purification the paraffin scales are melted in a large pan by the introduction of steam through a perforated iron coil. The mechanical impurities and water having subsided, the supernatant liquid is brought into another tank, where it is

mixed with a certain quantity of naphtha, and the mixture is allowed to settle for some time to remove adhering water and suspended matter. The clear liquid is run down into cooling tins, which are arranged in racks or frames. In these it is allowed to remain about 24 hours, and cools into firm cakes. These cakes are turned out, wrapped in canvas sheets, and pressed between iron plates in a hydraulic press. The naphtha, carrying with it oil and coloring matter, is squeezed out, leaving a dry cake of paraffin. This operation of solution in naphtha, cooling, and pressing is repeated two, three, or four times until the desired degree of purity is reached. The cakes are then melted up and steamed in the still until all traces of naphtha are removed. The steamed paraffin is carefully separated from water and run down into the so-called "black tank," where it is mixed with from 5 to 8 per cent. of freshly burned animal charcoal, and after vigorous stirring for half an hour allowed to settle. It is then run through filters to remove the fine particles of charcoal that refuse to subside, and cooled in pans or tins, when it will be ready for candle-making.

Several materials have been substituted for charcoal in the above process, such as the addition of about 12 per cent. of fullers' earth, at a temperature of 230° F. The mixture is well agitated, then left to settle, and the clear paraffin is run off. The fullers' earth may be cleansed from paraffin by washing or agitation, and used again. By another process, silicates of magnesia and of other bases may be employed for the same purpose. Moreover, a very ingenious process has been patented in England by Mr. Sterry, for removing the oil from paraffin without pressing. It consists in simply washing or rather kneading the paraffin with a solution of soap at a temperature of 9° F. below the melting point of the paraffin. The product is pure white, but opaque, hence its use is limited.

In this country paraffin is wholly a by-product in the manufacture of lubricating oils, or in the treatment of the heavy oils proceeding from the distillation of petroleum towards the last of the process.

For the following description of the manufacture of paraffin in

this country we are indebted to Benjamin J. Crew's Practical Treatise on Petroleum.

In treating the subject from the standpoint of the American refiner, the preparation of lubricating oils and the manufacture of paraffin-wax run so closely together that at some points at least they touch and must be treated as though they belonged to one subject. The manufacture of either or both begins when the residuum is placed in the still. The first products of this distillation down to about  $32^{\circ}$  B. (which when they are received into one tank constitute an oil of  $38^{\circ}$  B.) are returned as crude oil.

After the separation of this first part, the products of the still are pumped into the "paraffin agitator," where they are first treated with acid, and after drawing off the "acid sludge," washed with alkali and water. Care should be taken throughout that the proper temperature be preserved so that the paraffin shall be maintained in perfect solution. The oil is then allowed to flow by gravity or it is pumped into tanks, provided with a steam coil, in order that its contents may be preserved in a perfectly limpid state to permit of the settling of the water. This being drawn off, the contents are removed to another apartment, the temperature of which has been artificially lowered by a freezing machine, where it is subjected to the chilling process.

In the winter the ordinary temperature is sufficient to crystallize the paraffin. If the process is to be carried on during the warm weather, the contents of the tank are barrelled and the temperature of such a room may be reduced by a good apparatus to  $10^{\circ}$  or  $15^{\circ}$  F., even in very hot weather. An exposure of the paraffin-oil for forty-eight hours chills the whole mass to a complete solid. From the barrels the contents are shovelled out into small cotton bags of very strong material and subjected to powerful pressure by means of a hydraulic press. The cakes when removed from the bags are somewhat variegated in color, some portions being of a light lemon, others presenting quite a greenish hue. The contents of the bags are thrown into a steam tank, where the cakes are melted by live steam, one per cent. of soda lye is added, and the whole thoroughly steamed, the condensed water withdrawn, and when sufficiently cool to admit of the process, about 25 per cent. of benzene is added, and the whole vigorously stirred until



a homogeneous mixture is obtained. The contents of this tank are ladled out into shallow tin-pans, holding about 5 to 10 gallons each, which are allowed to remain in the cold room for three to four days. This product is again subjected to pressure in clean bags. The paraffin thus obtained is in large crystals with a slight tinge remaining, and having a much higher melting point (about  $130^{\circ}$  F.) than the crude article first described.

Chemically, paraffin is a mixture of hydrocarbons of the series  $C_nH_{2n+2}$ . It is white and odorless, soluble in alcohol, and can be distilled without decomposition. The melting and congealing points of the various kinds of paraffin vary very much, as well as the specific gravity. Sauerlandt gives, for instance, the congealing point of paraffin as from  $100.5^{\circ}$  to  $179.5^{\circ}$  F., and its specific gravity as from 0.869 to 0.943. The higher the melting point, the greater the specific gravity.

Pure paraffin is sometimes used alone for candle-making, but is generally mixed with proportions of hard stearic acid varying from 5 to 10 per cent.

*Ozokerite or mineral-wax.*—Ozokerite or mineral wax occurs generally in fissures and cavities in the neighborhood of coal fields or deposits of rock-salt, or under sandstone pervaded with bitumen. Although it is widely distributed over the world, a solid paraffin with a high melting point is only obtained from the deposit in Gallicia, where miners' candles have long been made from it. The color of the mineral varies from brown to greenish and yellow tints; its fracture is resinous.

The structure and melting point of ozokerite depend, according to Sauerlandt, on its principal constituents—paraffin and “waxy resin”—because, with the distillation of ozokerite conducted with superheated steam and the avoidance of decomposition, he distinguishes the following constituents:—

1. Liquid hydrocarbons with a low boiling point.
2. Paraffins, chiefly with a boiling point of from  $140^{\circ}$  to  $158^{\circ}$  F.
3. Resinous bodies, called “waxy resins.”
4. Bituminous resins.
5. Coke.

Paraffin and waxy resins are solid bodies, the latter having a higher melting point than the former.

The paraffin mass obtained by distillation has a melting point of from  $113^{\circ}$  to  $122^{\circ}$  F. After complete crystallization, the paraffin scales are separated from the oil by centrifugals and filtering presses. The scales thus obtained still contain 20 to 25 per cent. of oil. To separate this, the paraffin is packed in press-cloths, and subjected to pressure in a hydraulic press with heated plates.

Reichenbach's method of purifying the crude paraffin with concentrated sulphuric acid has been almost entirely abandoned, as also the distillation over lime or chloride of lime. The crude paraffin, after melting, is at present treated in a similar manner as petroleum-paraffin, with about 15 to 25 per cent. of benzene of not more than 0.785 specific gravity, and then subjected to pressure in a hydraulic press. The operation is repeated several times, according to the desired degree of fineness, and the benzene adhering to the paraffin after pressing expelled by treating with steam for ten to twelve hours. The paraffin is then fined by digesting with animal charcoal or similarly acting substances, and finally filtered through blotting paper. Ozokerite paraffin thus obtained shows a melting point of about  $144^{\circ}$  F.

The process of purification by acidification with strong sulphuric acid gives *ceresin*, a substance much resembling beeswax in consistency and fracture. By this method, the *whole* of the mineral is converted into a homogeneous yellow substance without much loss except that of filtration and a certain amount of charred products.

The process of preparing *ceresin* by means of sulphuric acid is, however, defective, and now almost entirely replaced by that of extraction, which yields a paler product from the start, which is easier to purify.

The specific gravity of *ceresin* is much lower than that of beeswax, it varying between 0.915 and 0.925 (beeswax 0.963 to 0.969) at  $59^{\circ}$  F. The melting point varies between  $154^{\circ}$  and  $176^{\circ}$  F., the congealing point being on an average  $37^{\circ}$  to  $39^{\circ}$  F. lower, while the melting and congealing temperatures of beeswax are the same.

Ceresin is extensively used as a substitute for wax. As a material for candles, it can, however, not be utilized by itself, as the candles do not burn with a clear flame like those from stearin, paraffin, or beeswax, but it can be advantageously used as an addition to those materials.

The use of ozokerite paraffin for the manufacture of candles has many advantages. It has a very high melting point, and does not bend or soften in a warm atmosphere. It has great illuminating power, burns with a dry "cup," and is not so liable to gutter as ordinary transparent candles. It is entirely free from smell, not unctuous to the touch, and has the appearance of the finest bleached beeswax.

### *Waxes.*

1. *Waxes of animal origin.*—The most important animal wax for the manufacture of candles is

*Beeswax* from *Apis mellifica*, Linn., or the bee, which yields most of the wax found in commerce. The wax is secreted upon the ventral scales of the bee, and used by the insect for the construction of the comb, the cells of which are hexagonal with angular bottoms. The comb, from which the honey is allowed to drip, is first subjected to pressure, and is then melted in boiling water, to free it from adhering honey and other impurities, and then poured into flat moulds, previously moistened, and left to cool.

In a pure state, beeswax is perfectly white, but becomes colored by the contact with honey and pollen, the color of crude wax being more or less yellow, and sometimes even reddish or greenish, according to the materials used by the bees.

For purification, the process of melting the wax in hot water is repeated until it is freed from all honey, and shows no longer a gray color. The destruction of the coloring matter by chemical means is not advisable, as chlorine and other bleaching substances exert a decomposing effect upon the wax, making it brittle and crumbling, which has to be remedied by an addition of tallow. The use of chlorine is further objectionable on account of the



formation of products which, later on, by combustion, develop hydrochloric acid.

The best method of bleaching is to melt the yellow wax in a large vat by means of steam. It is then run off, while in a melted state, into a trough, called a cradle, which is perforated on the bottom with holes, and placed over a large water-tank, at one end of which is a revolving cylinder almost immersed in water. By this means the wax is solidified, converted into a kind of ribbon, and conveyed on the surface of the water to the other end of the tank. These ribbons of wax are lifted out, and carried in baskets to the bleaching ground, where they are exposed to the air for one or two weeks, according to the weather, being turned every day, and watered from time to time.

By another process, the wax, purified by steam, is obtained in threads or grains by allowing it to drop in a thin stream, or drops, into ice-cold water from a revolving tinned-copper boiler provided with one or more cocks. By this granulating and thread-drawing process, a greater surface is exposed, and the bleaching process shortened at least one-third.

After exposure upon the bleaching grounds the wax is re-melted in hot water, strained, and poured into moulds to cool. The loss in weight caused by bleaching is from 2 to 10 per cent.

To assist the natural or sun-bleaching process, which is based upon the formation of ozone and the action of the latter upon the coloring matter, by a further formation of ozone, the yellow wax is melted together at a moderate heat with rectified oil of turpentine in the proportion of 8 parts of wax to 1 to  $1\frac{1}{2}$  parts of oil of turpentine. The mixture is melted, ribboned, and bleached in the same manner as wax. The bleaching process is much accelerated, being finished in five to six days, after which the odor of oil of turpentine has entirely disappeared.

White wax is tasteless and inodorous, translucent on the edges, brittle, not unctuous to the touch, softens at  $86^{\circ}$  F. and in the hand by kneading, melts at  $145.5^{\circ}$  to  $147^{\circ}$  F., and has a specific gravity of 0.965 to 0.969.

In buying wax great care should be exercised, as both the yellow and white varieties are frequently adulterated. The spe-

cific gravity and melting point are of special importance for the determination of quality.

The most common adulterations are as follows: *Admixture of water* to increase the weight, which is readily recognized by the dull and rough fracture and by slowly heating to the melting point and cooling, whereby the water separates, the quantity being determined by weighing the cooled wax. *Additions of pulverulent substances*, such as *yellow ochre, brick-meal, pea-flour, heavy-spar, clay, litharge, etc.*, are also detected by melting, whereby they separate. *Stearin, resin, and vegetable waxes* increase the specific gravity. Pure white wax floats in the centre of a fluid consisting of 2 parts of alcohol of 0.830 to 0.831 specific gravity and 7 parts of distilled water, even if the fluid is brought to a specific gravity of 0.965 to 0.970 by the addition of distilled water.

Pure yellow wax shows the same phenomenon in a fluid of 0.955 to 0.965 specific gravity, prepared as above from 1 part of alcohol and 3 parts of water. Adulterations with the above-mentioned substances would cause the wax to sink, while sophistication with paraffin, ceresin, ozokerite, etc., would make it float upon the surface. Generally speaking the principal adulterations are with yellow ceresin for yellow wax and white ceresin for white wax. Paraffin or ceresin, etc., is recognized by heating a sample of the suspected wax with fuming sulphuric acid in a porcelain dish. Pure wax is thereby entirely decomposed and converted into a black, jelly-like mass, from which the paraffin is separated in an unchanged form after cooling. *Tallow*.—By pouring spirit of sal ammoniac over scraped wax in a test-tube and heating, the fluid does not become turbid as is the case in the presence of tallow. *Stearic acid* is detected by dissolving a small quantity of wax in ten times the quantity of chloroform and adding lime-water. Pure wax remains dissolved, while in the presence of stearic acid a granular precipitate of stearate of lime (lime-soap) is formed. *Resin*.—Boil the wax with concentrated nitric acid; in the presence of resin the fluid acquires a reddish color. By adding water to the fluid after cooling, and freeing it from wax, it becomes turbid, and the resin separates as a yellow, flaky precipitate, which dissolves with a reddish-brown color in

caustic ammonia. *Japan-wax*.—Boil about equal parts of borax and wax with fifteen to twenty times the quantity of water; with pure yellow wax the milky, turbid mixture separates gradually into a clear, yellowish fluid and with pure white wax into a clear fluid, with the wax in both cases floating on top. In the presence of Japan-wax the whole remains milky and, according to the quantity of adulteration, thickly fluid or jelly-like and rigid.

There are a number of other methods of examining wax, but we have given the most simple and reliable ones and the quickest of execution.

*Chinese-wax* is produced by *Coccus pela*, Westwood, or the white-wax insect of China, upon the branches of *Fraxinus chinensis*, Roxb., or the Chinese ash. The rearing of the insect in China is now an industry next to silk in importance.

Chinese-wax, or insect-wax, differs entirely from Japan-wax, with which it is frequently confounded, especially in literature. The branches of the trees having become incrustated with the wax secreted by the insects, are cut off in the month of August and boiled in water, whereby the wax collects upon the surface. Later on it is remelted and poured into deep pans, where it cools to a transparent and very crystalline mass. The wax is pure white or of a very slightly yellowish shade, tasteless and inodorous, lustrous and crystalline throughout. In appearance it resembles spermaceti, but is much harder and more brittle, can be almost pulverized, and is of a more fibrous structure. It has a specific gravity of 0.970 at 59° F., melts at 179.5° to 181.5° F., yields little to alcohol and ether, but is very readily soluble in benzene. It is difficult to saponify with boiling potash lye. It is not a mixture of various substances, but consists almost entirely of ceryl cerotate  $\left. \begin{matrix} \text{C}_{27}\text{H}_{53}\text{O} \\ \text{C}_{27}\text{H}_{55} \end{matrix} \right\} \text{O}$ .

In China and Japan it forms an important article of commerce, being used in the manufacture of candles, which, it is claimed, possess ten times the illuminating power of ordinary candles. It is also used for coating candles manufactured from Chinese vegetable tallow. At the present time Chinese-wax is not of much commercial importance, as China consumes immense quantities,



but formerly it was largely brought to England in broad cakes perforated in the centre, and was much used in the manufacture of candles.

*Spermaceti*.—The main source of this beautiful candle material is the cachelot or sperm whale (*Physeter macrocephalus*). It is found not only in the “head matter” but generally diffused throughout the blubber. After the death of the animal it forms a crystalline mass. The whale-oil is strained off, the remaining crystalline mass pressed and treated with weak caustic soda or caustic potash lye to saponify any adhering oil. It is then rinsed off in water and remelted in boiling water.

Crude spermaceti, which is seldom found in commerce, forms finger-thick plates, which are yellowish, transparent, of a foliated structure, and a rancid, fishy odor. To convert it into the commercial article the plates are melted down and boiled for two or three hours with caustic lye of  $14^{\circ}$  B. in the proportion of 40 parts by measure of the former to  $1\frac{1}{2}$  of the latter. The mixture is kept at a low, equable temperature, and allowed to remain at a gentle simmer, while the soap that has been formed rises to the surface and is skimmed off. The heat is then raised to about  $250^{\circ}$  F., and the mass is treated with small successive doses of water, the additional scum being carefully taken off as it rises, till the whole is clear. It is then drawn off to crystallize in flat tin-dishes. The cakes thus obtained are again reduced to powder, which is wrapped in linen bags and subjected to hot pressure in a very powerful hydraulic press. The cakes of spermaceti coming from the press contain still some oil which has to be extracted by saponification, which is effected by boiling with strong alkaline lye at  $235^{\circ}$  F., removing the scum as before. Further purification is then effected by introducing a small quantity of water at intervals, while the heat is lowered. The supernatant spermaceti, now perfectly colorless and transparent, is cast into blocks and crystallized. Pure spermaceti is talcky to the touch, tasteless, inodorous, and friable. It has a specific gravity of 0.943 at  $59^{\circ}$  F., melts at between  $122^{\circ}$  and  $129^{\circ}$  F., and, like wax, congeals immediately below the melting point. It is a mixture of various fats, but not of glycerides. By recrystallizing it from spirit of wine, cetyl palmitate  $\left. \begin{array}{l} \text{C}_{15}\text{H}_{31}\text{O} \\ \text{C}_{15}\text{H}_{33} \end{array} \right\} \text{O}$  is formed.

Spermaceti is not readily adulterated, as by every admixture it loses in a high degree its peculiar and very characteristic properties. Adulterations are readily recognized by greater hardness, absence of nacreous lustre, and the small, foliated, crystalline structure. For the manufacture of candles, spermaceti is generally mixed with about 3 per cent. of wax or paraffin, to counteract its crystalline structure. The candles burn with a bright, inodorous flame.

The United States and England supply most of the spermaceti.

2. *Waxes of vegetable origin. Japan wax.*—This wax is obtained from the berries of several trees of the genus *Rhus*, indigenous to China and Japan. The usual method to obtain the wax is as follows: The berries are ground and the floury mass obtained is boiled with water in large kettles and the wax skimmed off. To remove foreign admixtures, the cooled wax is several times remelted until entirely clear. By another process, the berries are first beaten with bamboo flails, then dried for fourteen days, slightly roasted, and ground. The floury mass is packed in small bags and exposed to the action of water vapor to melt the fat in the cells, which is then expressed by means of various kinds of presses. The extraction process has recently also come into use. The expressed wax being somewhat greener in color than that extracted with ether or bisulphide of carbon, it is boiled with dilute lye, whereby it becomes granular and more susceptible to the bleaching process, then washed with water and exposed to the sun. According to the degree of bleaching, the wax is known as prime and second.

Japan-wax is now brought into commerce in square cakes weighing about 140 pounds each. It has a waxy appearance, is whiter and more brittle than beeswax, and breaks even or largely conchoidal. The surface of a fresh fracture is lustreless, while that of a cut one has a wax-like lustre. The wax can be kneaded between the fingers and chewed to a powder. Its odor and taste are somewhat resinous, tallowy, and rancid. By storing, it becomes yellow to brownish, and, as it contains much water, is covered with a white, dust-like efflorescence.

Japan-wax melts at 128° to 130° F. and congeals at 105° to 106° F. If remelted a short time after congealing, it melts at

107.5° F., and reacquires the ordinary melting point of 128° F. only after long storing. The specific gravity of the crude wax is 1 to 1.006, and that of bleached wax 0.970 to 0.980 at 59° F.

In Japan the wax is used as a substitute for tallow and beeswax in the manufacture of candles, and for producing a lustre upon turned-wood articles. In Europe it is employed in the manufacture of wax-matches, and as an addition to bleached beeswax in the manufacture of candles, as it facilitates the removal of the latter from the moulds. The principal European market is London, which, in 1881, imported over 11,000 boxes, of about 132 pounds each.

*Fig-wax* or *getah-wax* is obtained by evaporating the milky juice of *Ficus cerifera*, Blume, or the wax-fig tree, indigenous to West and Central Java, Sumatra, and Ceylon. It has a reddish-brown color, but becomes pure white by bleaching. It is very brittle, softens at 113° F., and melts at from 133° to 134.5° F. It is used like beeswax in the manufacture of candles.

*Cow-tree wax*.—By making incisions into the trunk and limbs of the cow-tree (*Galactodendron utile*), indigenous to the mountains of South America, a milky juice resembling cow-milk in color and taste is obtained. It contains 30 to 35 per cent. of wax, which is separated by boiling. The wax is somewhat transparent, can be kneaded, melts at from 122° to 125.5° F., and can be partly saponified. In external appearance, it resembles beeswax more than any other vegetable wax, and yields very good candles, which burn with a bright flame.

*Myrica wax* or *myrtle tallow*.—The fruits of *Myrica cerifera*, Linn., a fragrant shrub growing near Lake Erie, but chiefly along the Atlantic coast of North America, secrete a wax which is deposited in thin layers, and covers them with a snow-white crust, interspersed with small brown or black specks. The fruits are boiled in water, and the fatty mass collecting on the surface is skimmed off, and poured into shallow vessels. The yield is about 20 to 25 per cent., or 2 pounds of wax from 8 to 10 pounds of berries.

The wax has a deep-green color, due to chlorophyll, which, by exposure to light and air for a long time, changes to gray-yellow, but only a few millimetres below the surface. It breaks with a



shallow, conchoidal fracture. Its specific gravity is 1.000 to 1.005 at 59° F., and it melts at 113° to 115° F. It is used like beeswax, but, being less ductile and plastic, is generally mixed with it. In this country it is much liked for the manufacture of candles, as they diffuse an agreeable odor when extinguished.

*Carnauba-wax* is secreted in thin films and scales on the leaves, stalks, and berries of a Brazilian palm, *Corypha cerifera*, Linn. By shaking or pounding the leaves, etc., the wax peels off, and is melted over an open fire, or boiled with water, whereby the foreign substances settle on the bottom, and the wax collects on the top. The wax is dirty gray-yellowish to greenish, hard, brittle, and can be readily rubbed to a powder. It is tasteless, and, when fresh, smells like new-mown hay, but later on becomes inodorous. It is purified by remelting, and has then a greenish color which cannot be removed.

Carnauba-wax acquires a reddish color by boiling with potash lye, is only partially and with difficulty saponified with alcoholic potash lye, and consists of myricyl alcohol,  $C_{30}H_{62}O$ , ceryl alcohol,  $C_{27}H_{56}O$ , and cerotic acid,  $C_{27}H_{54}O_2$ , besides an undetermined quantity of resin. According to Allen and Thomson, 54.87 per cent. of it is non-saponifiable.

Fresh, purified carnauba-wax melts at 185° to 187° F., old wax at 194° to 196° F., and congeals at 187° to 188.5° F., the cooled mass being somewhat crystalline. In melting, the wax diffuses a slight but not disagreeable odor, and by dry distillation yields a paraffin-like product. Its specific gravity is 0.995 to 0.999 at 59° F.

Valenta has determined the melting points of mixtures of carnauba-wax with stearic acid, ceresin, and paraffin:—

Addition of carnauba-wax with a melting point of 185° F. Per cent.	Mixtures of carnauba-wax with		
	stearic acid, with a melting point of 137° F.	ceresin, with a melting point of 163° F.	paraffin, with a melting point of 140° F.
5	157.5°	174.3°	165.0°
10	164.75	177.0	174.3
15	166.15	179.0	178.0
20	167.0	180.5	179.0
25	168.2	181.4	179.0

Carnauba-wax is extensively used in the manufacture of candles and wax-varnish.

*Palm-wax* is obtained from the common wax-palm (*Ceroxylon andicola*) and the Klopstock palm (*Klopstockia cerifera*). The trunk of both varieties secretes a resinous wax, which is scraped off, and converted into a compact mass by melting over an open fire, and purified by remelting. By another process the bark of the trees is boiled in water, whereby the wax, however, does not melt, but only softens, while the admixed impurities settle on the bottom.

The wax thus obtained presents a yellow or yellowish-white mass, and is brought into commerce either in irregular lumps or balls. In hardness and brittleness it corresponds to carnauba-wax. It breaks with a conchoidal fracture. Both the crude and purified waxes have a specific gravity of 0.992 to 0.995 at 15° F., and, though they become soft by the heat of the hand, melt only at 215.5° to 221° F.

Palm-wax is a mixture of wax and resin from which, by dissolving in much alcohol, the wax is separated as a white, somewhat crystalline jelly. By repeated washing and recrystallizing from a large quantity of boiling alcohol, it is entirely freed from resin. This pure wax is white-yellow, resembles beeswax, and melts at 161.5° F. to a but slightly colored liquid. It consists of ceryl cerotate and myricyl palmitate with resin. It is used in the manufacture of candles mixed with a little tallow to make it less brittle. The so-called palm-candles have almost a lemon-color, as palm-wax cannot be bleached.

There are other waxes much used in their native countries, as Ocuba-wax from *Myristica bicuhiba*, Balanaphore-wax from *Langsdorffia hypogæa*, both growing in Brazil; also Andaquies-wax, Cuba-wax, and others of uncertain origin.

*Sebacylic acid* should receive some notice as a material for the manufacture of candles, especially as it might serve to impart to candles with a low melting point a higher one. It is obtained by the dry distillation of oleic acid, and by treating castor-oil or ricinoleic acid with soda-lye. From 100 parts of ricinoleic acid over 81 parts of sebacylic acid are obtained, which melts at 260.5° F. Added to stearic acid, it prevents it from crystallizing, and 1 to 5 per cent. of it mixed with readily-melting fatty substances imparts to them the hardness of wax.

## CHAPTER III.

## MANUFACTURE OF CANDLES.

*Wicks and their preparation.*—The wicks for candles require close attention, for it is essential that they should be of the right size, of uniform thickness, and free from loose threads and knots, the latter causing the candles to gutter. At present the wicks generally consist of twisted or plaited cotton threads, though sometimes flax or hemp is used.

The cotton-yarn most frequently employed is slightly twisted mule-yarn, Nos. 16 to 20 for tallow-candles, and Nos. 30 to 40 for stearin-candles, etc. The style of designating the degree of fineness of yarns is according to the English system, which is recognized in the United States, Germany, Belgium, Switzerland, etc. In order to understand the system, the following will suffice: Yarns, as is well known, come into commerce in hanks or skeins. The reel, upon which they are produced, has a circumference of  $1\frac{1}{2}$  yards or 54 inches, English measure; 80 threads form a lay or wrap, 7 of which constitute a hank, the latter representing, therefore, a thread length of  $54 \times 560$  English inches, or 2520 feet. The number of the yarn simply indicates the number of hanks which make an English pound weight, No. 16, for instance, being a yarn requiring 16 such hanks to make one pound, No. 40, 40 such hanks, and so on. In Austria and France other modes of numbering are in vogue, the numbers of an equal fineness, according to the Austrian system, being obtained by dividing the English number by 1.22, and that of the French mode of designation by dividing the English number by 1.18.

The thickness of the wicks varies very much and depends on the material as well as on the diameter of the candles. For tallow-candles twisted wicks are generally used, the following proportions being the most customary ones:—



For an 8 candle to the pound 42 threads of No. 16

"	7	"	"	45	"	"	16
"	6	"	"	50	"	"	16
"	5	"	"	55	"	"	16
"	4	"	"	60	"	"	16

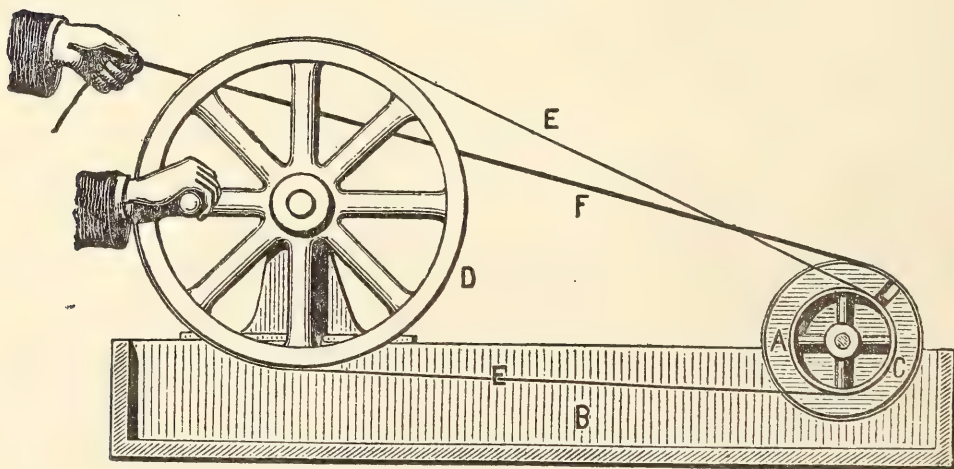
For stearin, paraffin, spermaceti, and many composite candles, plaited wicks of a much finer grade yarn are used, the following being customary :—

For an 8 candle to the pound 63 threads of No. 40

"	6	"	"	87	"	"	40
"	5	"	"	96	"	"	40
"	4	"	"	108	"	"	40

Before use the wicks, except those for tallow-candles, undergo a certain preparation by steeping them in the so-called "wick-mordants," by means of which they are rendered less combustile and the charred piece is prevented from swelling up. For this purpose compounds composed of solutions of ammoniacal salts (ammonium chloride, phosphate, etc.), of bismuth, of borates, or, boracic acid, are used. Bolley recommends a solution of ammonium chloride of 2° to 3° B. as being simple and cheap. Very

Fig. 101.



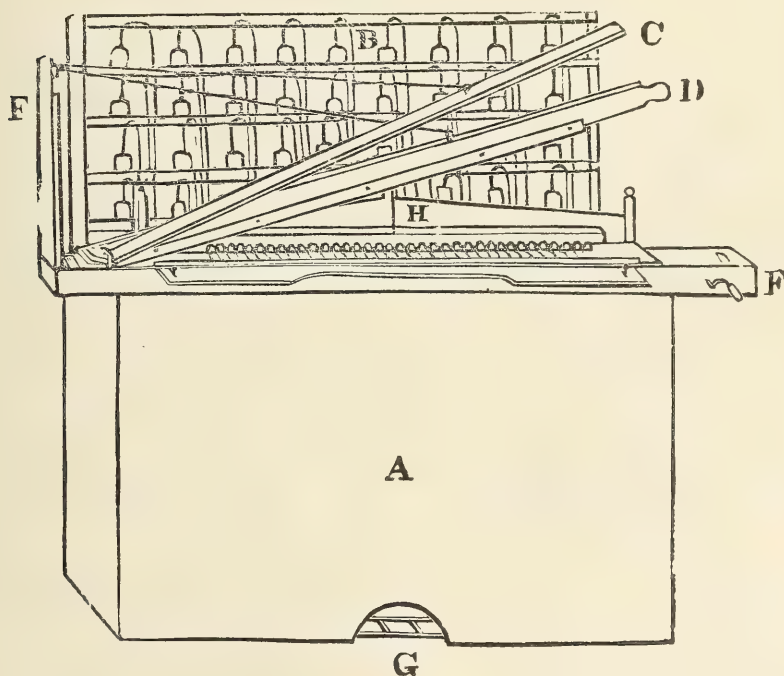
suitable also is a solution of 2 pounds of boracic acid in  $1\frac{1}{3}$  gallons of water. The wicks after thorough saturation with the solution are pressed and the excess of water removed by means of a centrifugal. Payen recommends an addition of a small quantity of

sulphuric acid to the solution of boracic acid. Sodium borate cannot be used on account of the strong coloration the flame acquires therefrom.

The twisting of the wicks is best effected by placing the single threads next to each other upon a table and turning so as to form a very elongated spiral. For the manipulations required many kinds of machines have been constructed, a very simple and efficacious one being shown in Fig. 101.

The circumference of the wooden cylinder *A*, which is the principal portion of the apparatus, is equal to the length of a wick, and several cylinders with different circumferences for the various kinds of candles can be used. The iron axle of the cylinder rests in a very simple bearing in the parallel sides of a box *B*, which are as wide as the cylinder *A*. On the outside of the cylinder is a pulley, *C*, connected with the driving-wheel *D*,

Fig. 102.



by means of a cat-gut cord, similar to a spinning-wheel. The workman sets the driving-wheel and the pulley in motion by means of a crank with one hand, while with the other he allows the wick to run upon the cylinder *A*. (The apparatus can also

be so arranged as to be worked by a treadle.) When a few layers of wick have been thus wrapped around the cylinder, the latter is taken from the apparatus and replaced by another. The wick upon the cylinder is then cut with a sharp knife, thus separating it into equal lengths.

A very handy wick-cutter for a large business, which is extensively used in this country, is shown in Fig. 102. *A* is the body of the machine inclosing the pulleys and other appendages that regulate the movement of the carriage *B*, which is set in operation by the treadle *G*. The carriage *B* rests upon the body; it is a kind of framework running on wheels, and containing a number of boxes placed shelfwise, and serving as a receptacle for balls of cotton-wick, the ends of which run through a notched reed below *H*, and come forward upon the twisting-board *E*, at the back of which a knife is fastened, that serves as the under blade of the movable clipper *D*. This, when drawn down vertically, severs the wick evenly. The twisting-box *EC* consists of two boards hinged, and moving on rollers. A turn of the crank near the end twists the wicks after they have been cut by the knife *D*, which having effected its purpose is drawn up again by a counterpoise *F*. At the front is a sliding-board so fixed as to regulate the length of the wicks.

Fig. 103 represents a plan of a machine for cutting wicks, known as Sykes's patent, and Fig. 104 a sectional elevation of

Fig. 103.

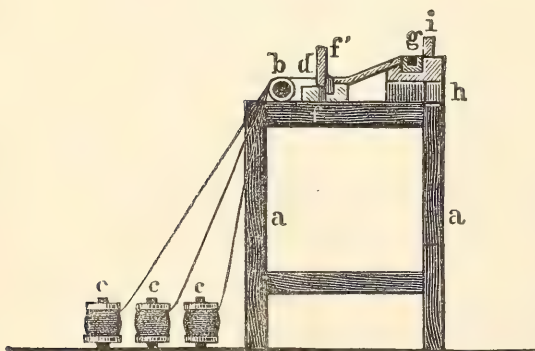
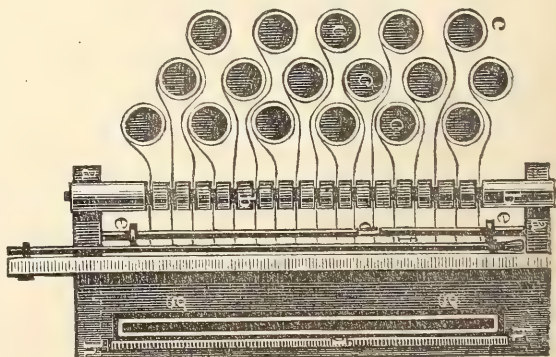


Fig. 104.



the same. *a a* is the frame, *b* a grooved roller over which the cords of twisted or plaited wicks are passed from the bobbins *c c c*, *d* is the clip or holder by which they are kept together. Fig. 105



represents the clip on an enlarged scale in a side view. It consists of two side bars *d d*, which towards the centre are somewhat thinner. On each side a steel spring is affixed, which keeps the bars somewhat apart from each other, and a clamp *e*, through

Fig. 105.



which, when it is moved towards the centre, the bars can be brought near. If the wick ends are to be kept together between the two bars, the clamps *e* must be pushed towards the centre. By taking the wicks out or placing them in they are pressed towards the end. In the rear of this clip is a cutting apparatus consisting of a stationary blade, and a knife *f* which has a handle moving on hinges, *g* is a small trough filled with liquid fat, which may be kept in a fluid state by steam, *i* is a band resting upon the table *h*.

The following is the manner in which the apparatus is worked: The wicks being prepared in suitable bobbins are brought down through the roller *b*, and secured between *d d* of the clip. The bars *d* and *d* are made to lay firm hold of the wicks, by moving the clamps *ee*, which bring them together in consequence of the wedge-shaped form of the upper bar *d*. The wicks being thus secured, the clip is lifted up and drawn forward by the workman, after which the free ends of the cotton that are left projecting from it are immersed or brushed over with the hot fat contained in *g*, and then laid on the top side of the square broach or suspending rod *i*, and are made to adhere to it with sufficient firmness to sustain them during the process of dipping by a slight pressure. Next, the clip is slackened by moving the clamps *e* outwards; they are then pushed forth over the cotton towards the bobbins, till the length to be cut for the candle is gained, when, by reversing the movements of the clamps *ee*, the wicks are again tightly grasped, and finally the clip is rested upon the table *h*, which is about one inch from the cutting apparatus. The movable blade *f* of the cutting apparatus is brought down, and the set of wicks of the proper length cut off, leaving as

much of the cotton adhering to the end next the suspending rod *i* as will support the next batch as before. The rod *i*, with the wick adhering to it, is placed in the dipping frame, and another rod again loaded with wicks as before, and so on till the frame is full.

The bars of the clip may be hinged, so that, when open, they will allow the bundle of yarn or cotton to pass freely, but, when closed, they will take a firm hold of them, as shown in Fig. 105.

A great many devices in plaiting and gimping of wicks have been patented and used, but it has narrowed down to the simple plaited wick for nearly all moulded candles, and the coarse, twisted one for dipped candles.

*Dipped candles.*—No doubt the oldest method of manufacturing candles is by dipping, *i. e.*, by repeated immersing of the wicks in melted fat until the required thickness is obtained. Dipped candles are always made of tallow more or less purified. By mere melting and straining, the tallow is, however, not obtained entirely free from admixture of fine undissolved substances. For separating these substances, therefore, it must be clarified. This is done by remelting it upon water, either over an open fire or by steam. Generally, no more water than 5 per cent. is taken, and stirred well with the tallow, till the mixture forms an emulsion. The whole is then allowed to rest, without further heating, till the water has separated, when the tallow may be drawn off or ladled off. Sometimes, in order to conceal the yellowish tint, a very little blue color is added to the clear fat, consisting of indigo rubbed finely with some oil, of which a few drops are sufficient even for large quantities. The process of clarifying is occasionally repeated.

To harden the tallow, add with constant stirring to 1000 parts of melted tallow, 7 parts of sugar of lead previously dissolved in water. After a few minutes, the heat is diminished, and 15 parts of powdered frankincense with 1 part of turpentine added, with constant stirring of the mixture. It is then left warm for several hours, or until the insoluble substances of the frankincense settle to the bottom. The hardening is produced by the sugar of lead, while the frankincense improves the odor of the tallow.

For carrying on the manufacture of dipped candles on a small

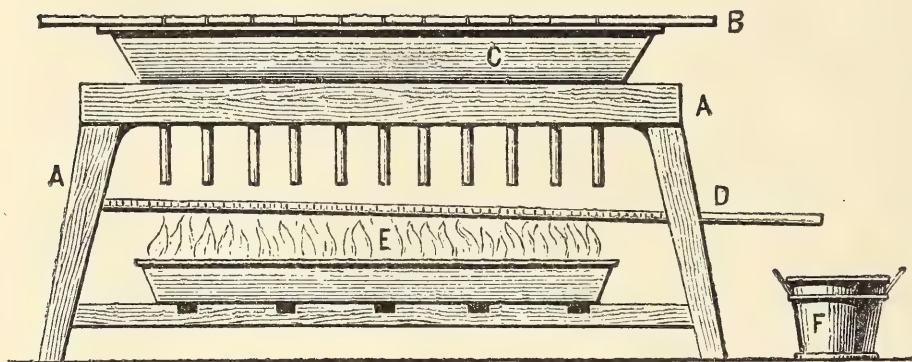
scale, but very simple apparatus is required. The clarified and remelted tallow is poured into a tightly joined trough three feet long by two feet wide at the top, gradually diminishing to three or four inches at the bottom. A handle is fixed on each side for its easy removal, and when not in use, it is closed with a cover. The operator commences by stringing 16 to 18 wicks at equal intervals on a thin wooden rod, about  $2\frac{1}{2}$  feet long, and sharpened at the ends. He then takes 10 or 12 such rods, and dips the wicks rapidly into the fluid tallow in a vertical direction. The tallow should be very liquid in order that the wicks be soaked as uniformly as possible, it being sometimes necessary to repeat the operation. On account of the high temperature of the tallow used, the wick, before cooling, has a chance to straighten out by its own weight. The workman then places the rods with the wicks in a certain order upon a frame to drain off. All the wicks being saturated with tallow, the second dipping is commenced by taking two or three of the rods with wicks on which the tallow has first congealed, and immersing them in the tallow, which in the mean while has somewhat cooled off, and commenced to congeal on the edges of the trough. By this means, a thicker layer of tallow than the first adheres to the wicks. The dipping is then repeated until the candles have acquired the desired thickness, they being, of course, returned to the frame after each dipping to cool. The conical spire at the upper end is formed by immersing *deeper* at the last dip. Should the tallow become too cold during the operation, it is restored to the correct temperature by the addition of hot tallow. Frequent stirring is also required to prevent the stearin and palmitin from crystallizing out. In order to be able conveniently to regulate the temperature, it is best to use a jacketed copper kettle in place of the wooden trough.

Dipped candles being seldom symmetrical, and generally somewhat thicker on the lower end, this defect is remedied by repeatedly dipping the lower end for a short time into hot tallow. Sometimes the candles are equalized by melting off, as shown in Fig. 106. The candles, strung on the rod *B*, are suspended in a trough without a bottom, *C*, so that the ends touch the copper or iron-pan *D*, which is somewhat inclined forward, and heated by



a coal-fire *E*. The tallow melted off runs into the collecting vessel *F*.

Fig. 106.



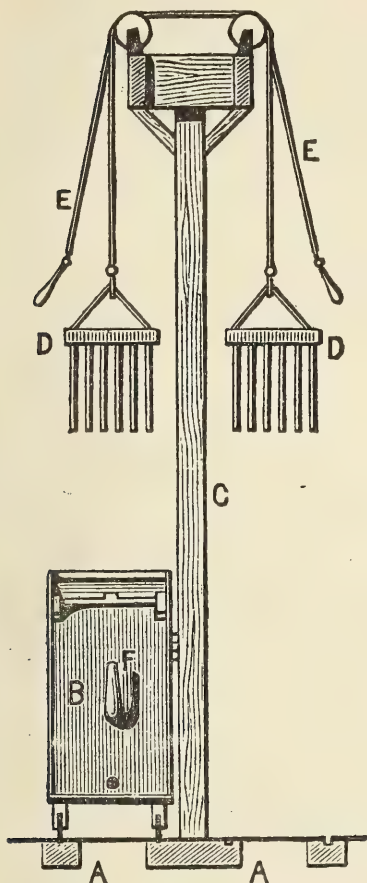
*A*, frame ; *B*, rod with candles ; *C*, trough without bottom ; *D*, pan ; *E*, coal-fire ; *F*, collecting vessel for fat.

By another method the candles are made symmetrical by passing them through a drawing plate. This is made of hard wood, 12 inches long, 2 or 3 inches wide, and about  $\frac{3}{4}$  inch thick. It is perforated by a number of holes, graded from large to small, the last being the size required for the finished candle. The holes have a slight bevel that the cutting-edge may be the sharper and the candles may run through with greater ease. The workman draws the candles first through the larger hole, which takes off a portion, then through a smaller one, which removes more, and so on until the desired size is obtained for the finished candle. This operation improves the appearance as well as the burning of the candle.

For dipping on a large scale there are some very convenient arrangements, as, for instance, a number of rods strung with wicks being arranged in a frame, which, by means of mechanical contrivances, are carried over pans in order to dip the wicks into the tallow and lift them out. Two methods are in use. The frames containing a number of rods with wicks are either suspended to supports standing alongside each other, as shown in Figs. 107, 108, and 109, and a trough filled with tallow is pushed backward and forward upon rails below the frames, or there is a

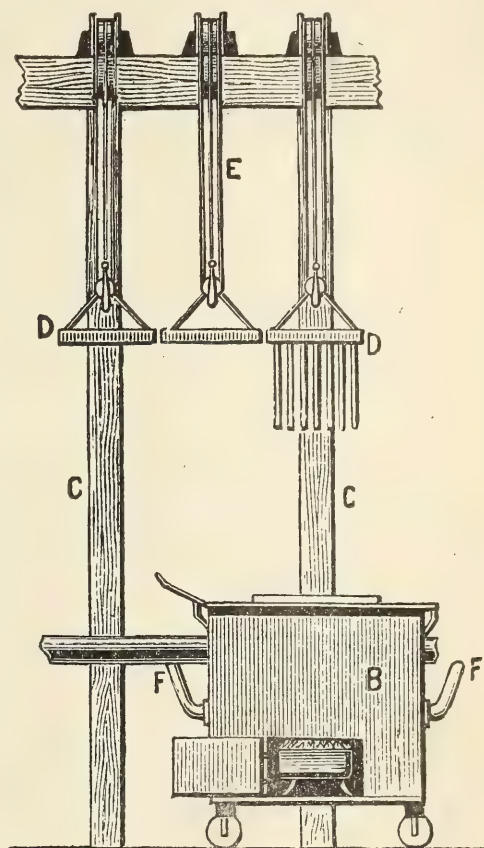
stationary tallow trough, as shown in Figs. 110 and 111, and the frames are advanced by means of a horizontal wheel on a vertical

Fig. 107.



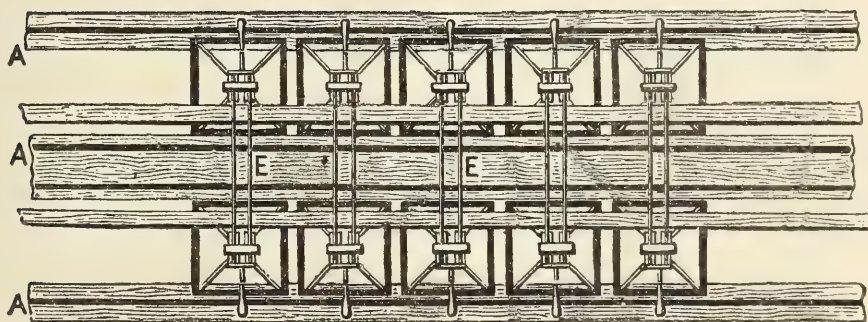
View.

Fig. 108.



Side View.

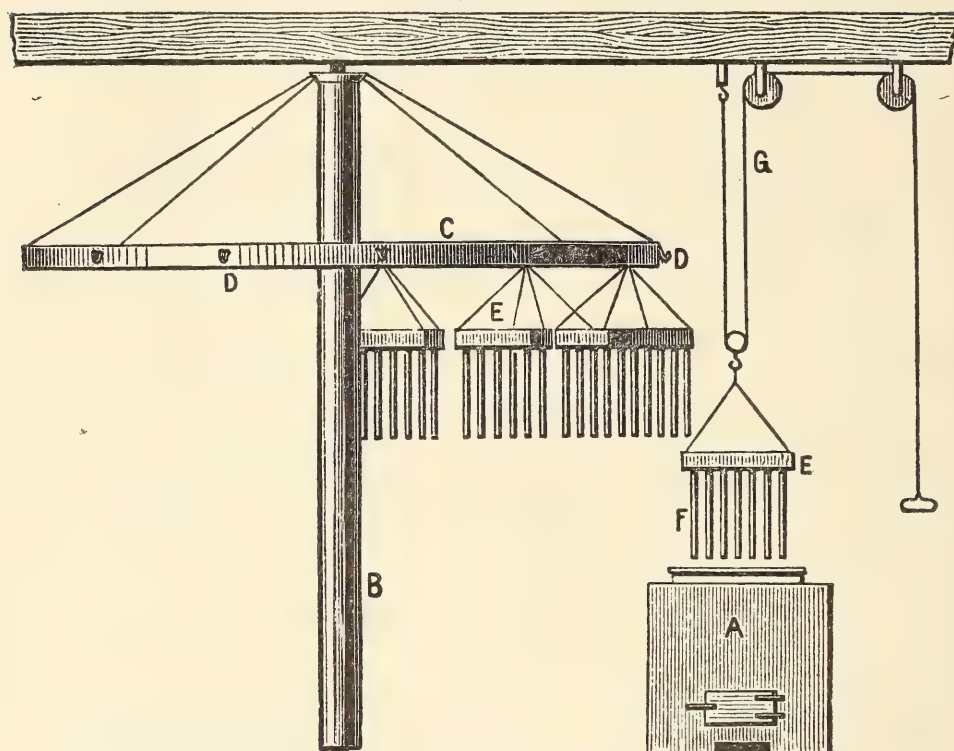
Fig. 109.



Ground-Plan.

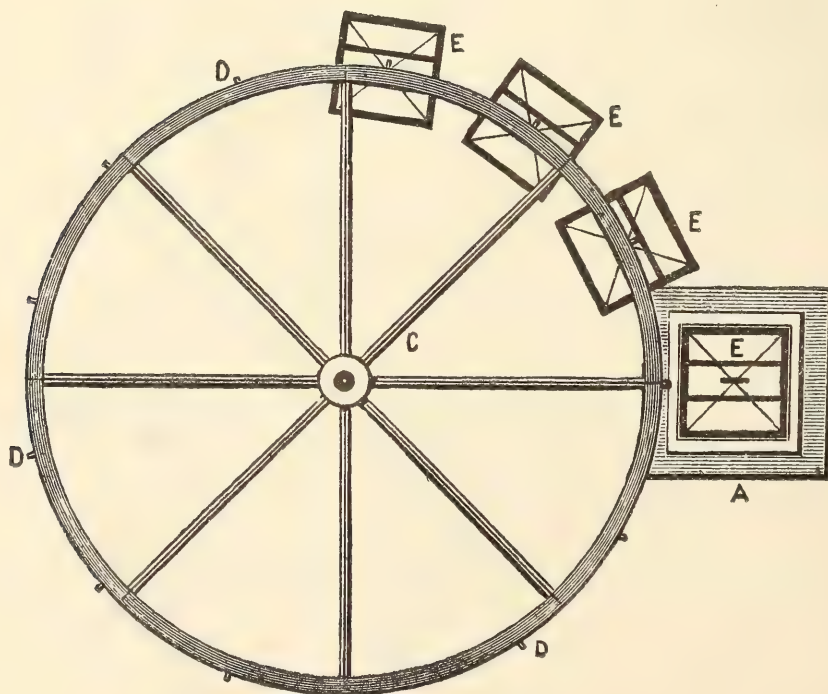
*A*, rails; *B*, transportable furnace with lard-trough; *C*, support with two frames; *D E*, ropes with handles for pulling the frames up and down; *F*, handles for moving the furnace.

Fig. 110.



View.

Fig. 111.



Ground-Plan.

*A*, lard-trough in a furnace; *B*, vertical shaft with horizontal wheel; *C D*, hooks; *E*, frames; *F*, wicks; *G*, ropes for lowering and raising the wicks.



shaft. The mode of operating these machines will be readily understood from the illustrations and explanations of the letters.

*Moulded candles.*—By far the greater number of candles now manufactured are moulded, by which they acquire a much more finished appearance. The operation of moulding is performed by stretching the wick in the axis of a nearly cylindrical mould and pouring the liquid material around it. The moulds should be of a material which is a good conductor of heat and does not absorb fat. They were formerly made of cast-iron, sometimes lined with pewter, of tin, brass, and even glass, but at present an alloy of two parts tin and one part lead is almost exclusively used for their construction. The moulds are narrow, somewhat conical, tubes, highly polished internally in order to impart a smooth surface to the candle. They are bored out by machinery so that the interior shall be perfectly true. They vary somewhat in form, but as a rule each mould consists of two parts, the body *B* and the head-piece *C*, fitting into the body as shown in Fig. 112. The head-piece is sometimes provided with a small stay, as shown in Fig. 113.

Fig. 112.

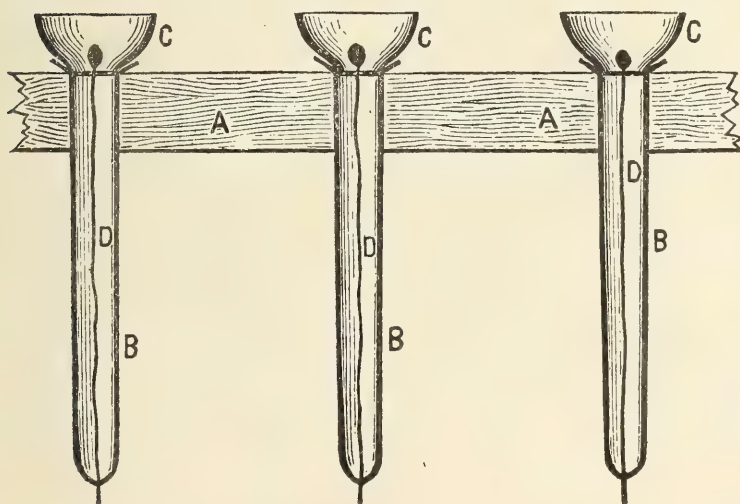
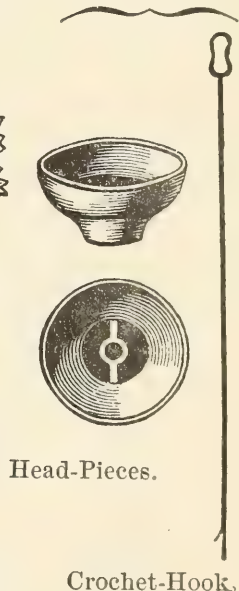


Fig. 113.



Head-Pieces.

Crochet-Hook.

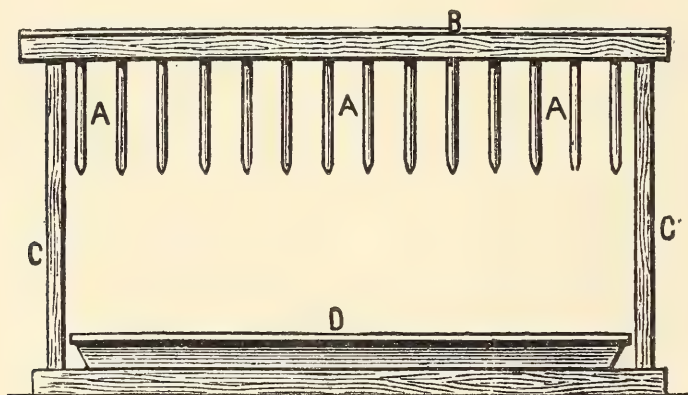
*A*, plate of the moulding table ; *B*, moulds ; *C*, head-piece ; *D*, wick.

The moulds made in this country are of a better form, and they are burnished by a vertical instead of a rotary motion,

which makes the candles easier to remove. The wicks are fixed in the longitudinal axis of the moulds by means of a long crochet-hook (Fig. 113), and secured by a peg at the "tip" and a cross-wire at the "butt" end of the candle.

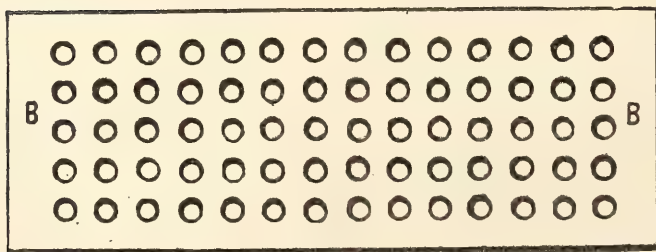
In moulding tallow-candles the temperature of the fat is of greater importance than in dipping, as it exerts an influence upon the expansion of the mould, thereby making the removal of the candle difficult. This danger appears when moulding too hot, while with the tallow too cold, bubbles and cavities are formed around the wick. The tallow should not be poured into the

Fig. 114.



View.

Fig. 115.



Ground-Plan.

*A*, moulds ; *B*, iron plate ; *C*, frame ; *D*, sheet-iron trough.

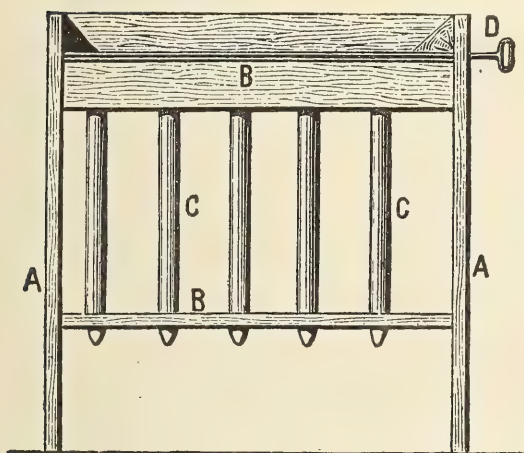
moulds until a thin film has formed upon the surface, which is an indication of the tallow commencing to congeal.

For moulding by hand a number of moulds with head-pieces

are suspended in holes of a frame, *C* (Figs. 114 and 115), covered with an iron plate, and on the bottom of which is a sheet-iron trough, *D*.

According to an American method, the moulds without head-pieces are fixed in an iron plate, *B* (Figs. 116 and 117), which is secured to a wooden frame, *A*, provided with a bottom. The wicks are suspended to a wire, *D*, and the iron plate, *B*, is surrounded on three sides with stationary ledges, while that on the fourth side can be taken away in order to remove the fat from the table after moulding.

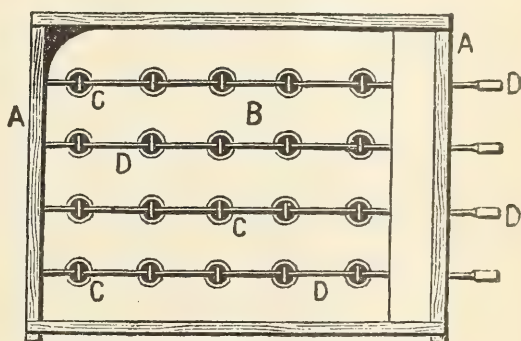
Fig. 116.



View.

*A*, frame ; *B*, iron plate ; *C*, moulds ; *D*, wire.

Fig. 117.



Ground-Plan.

After cooling, the candles are removed from the moulds and trimmed. This is best effected by means of a cutting apparatus, as shown in Figs. 118 and 119. The working of the apparatus will be readily understood from the illustrations and letters.

The tedious method of moulding by hand has been almost everywhere superseded by moulding by machinery. A very conveniently-arranged machine for moulding tallow-candles is shown in Figs. 120 and 121. The moulds *A* are fastened by means of screws to a table upon which the plate *C*, suspended by chains, can be lowered by means of the windlass and rope *B*. The mould-openings in the plate communicate with the funnels for the tallow. The wicks being strung upon rods, fluid-tallow is poured upon the plates to fill the moulds. When the candles



Fig. 118.

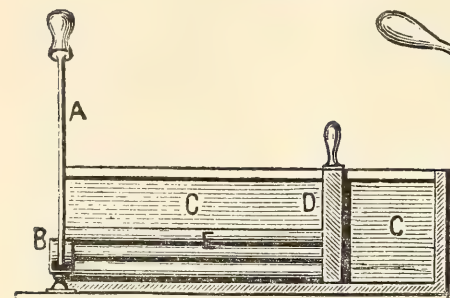
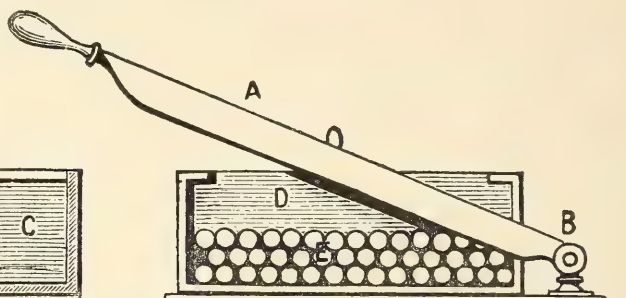


Fig. 119.



Cutting Apparatus.

Longitudinal section.

View.

*A*, knife ; *B*, hinge ; *C*, box ; *D*, measuring board ; *E*, candles.

are sufficiently cooled, the plate is drawn up, and the candles cut off by means of a large hinged knife. They are then allowed

Fig. 120.

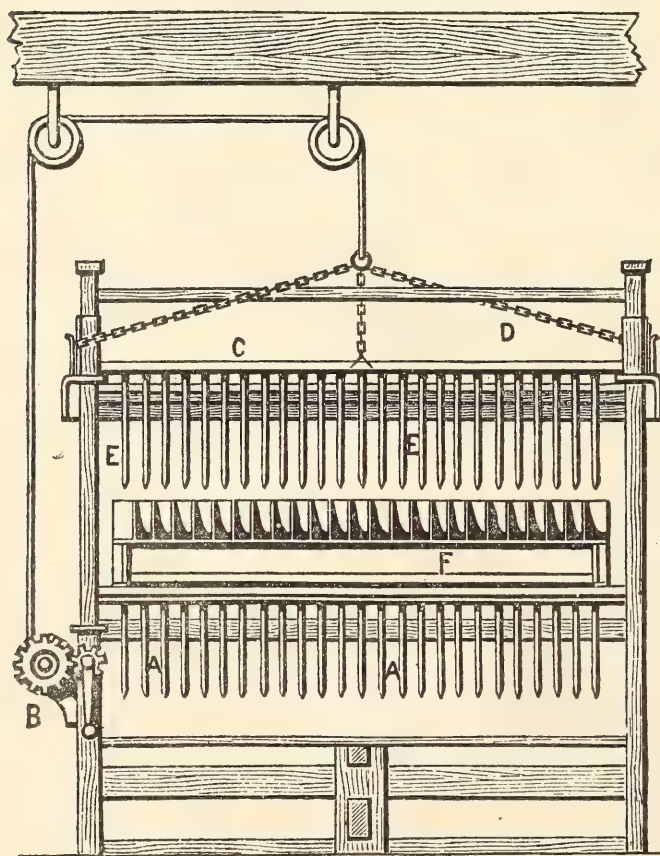
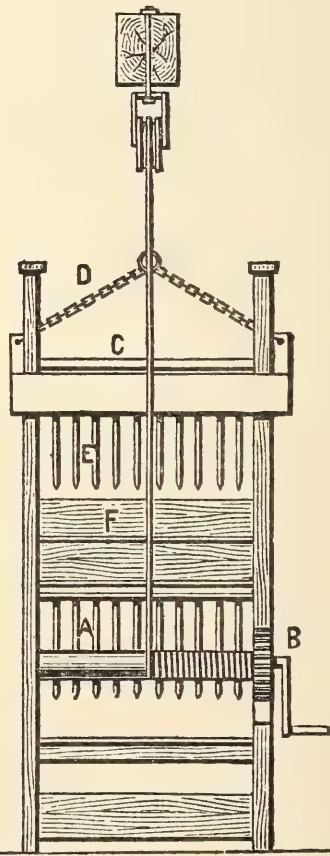


Fig. 121.



Moulding Machine for Tallow Candles.

View.

Side View.

*A*, moulds ; *B*, windlass ; *C*, plate ; *D*, chains ; *E*, finished candles ; *F*, box.

to drop into the box *F*, which is provided with corresponding holes, and covered to protect the candles from dirt.

*Morgan's moulding machine*, used in England for tallow candles, is so constructed that, with a sufficient number of stands, the moulding can be continued for an indefinite period, and at a saving of labor and time. The wicks in this machine are threaded through the moulds at the same time and by the same action as that which expels the candles. Fig. 122 shows the end elevation, and Fig. 123 a front view; Fig. 124 the plan, and Fig.

Fig. 122.

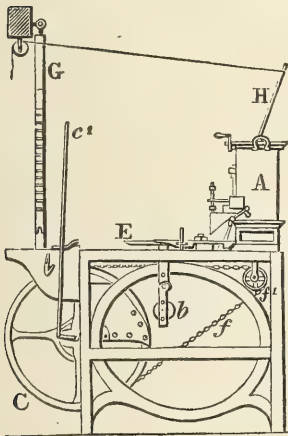
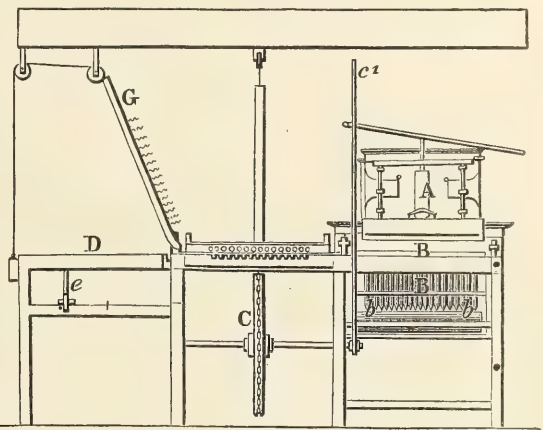


Fig. 123.



125 the elevation of the back opposite Fig. 123. *A* represents the vessel or reservoir containing the fat; *B* a series of moulds.

Fig. 124.

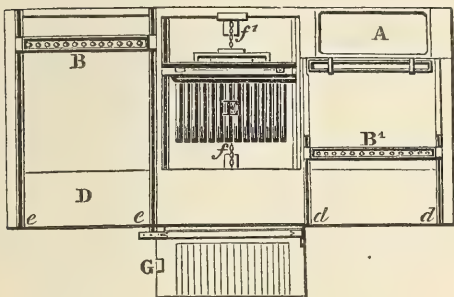


Fig. 125.

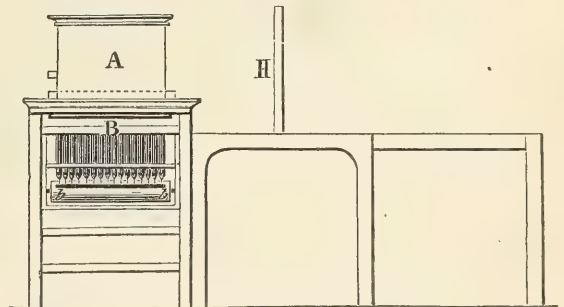


Fig. 126 shows the range of moulds constructed in a peculiar manner. Fig. 127 shows the upper end of one of these moulds, and Fig. 128 a plan view of the same. It will be seen that the top consists of several pieces: *b¹* is a portion of the cylindrical

side of the mould;  $b^2$  the movable portion. This latter  $b^2$  is hollow for the passage of the wicks, and fits closely to  $b^1$  when the tallow is poured in. But as soon as the candle is cold, and in a condition to be removed, instead of being drawn out in the

Fig. 126.

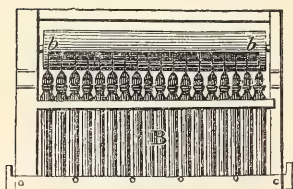


Fig. 127.

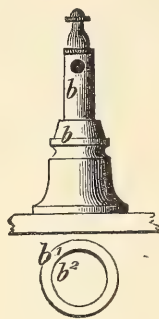
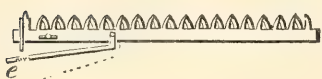


Fig. 128.

usual way, it is by this apparatus forced out by pressure applied to the extremity of the part,  $b^2$ , which, following the course of the candle as it is forced from the mould, rewicks the mould for another candle. In Fig. 126 is shown a hollow cylinder  $bb$ , holding the bobbins of wick revolving on a shaft passing through its length. Fig. 129 exhibits a series of nippers opening and

Fig. 129.



shutting by the action of the lever  $e$  holding the wicks (at the end and opposite to that of its entrance) in  $b^2$  in a perpendicular position.

To work this apparatus, we suppose a frame of moulds  $B$  regularly wicked and in the position at  $B^1$ , Figs. 123, 124, 125, where the case is supported perpendicularly on the small straight edges of a railway,  $dd$ , Fig. 124. In this position, they are run forward until they come under the reservoir  $A$ , when the tallow is applied in the usual manner. The moulds being filled are run along the railway  $dd$  to harden. When the candles are perfectly congealed, the moulds are brought to the position shown at  $B$  (Fig. 124), when they are placed on a railway similar to that shown at  $dd$  on the other side of the machine. Here they are pushed forward until they arrive at the hanging table  $D$ , which vibrates on the joint  $ee$ , and is then let down, but immediately returned to the longitudinal position



given at *D*, Fig. 124. The moulds *B* are moved until they arrive at the series of rammers *E*, as separately shown in Fig. 131, where the cylindrical case *bb* is removed by turning the jointed frame, as seen in Fig. 132, to be out of the way of the

Fig. 130.



Fig. 131.

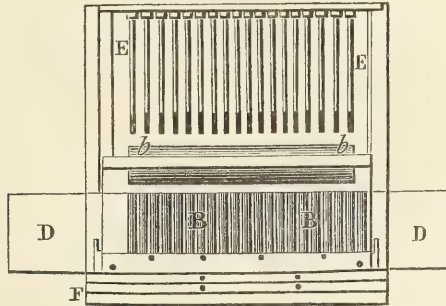
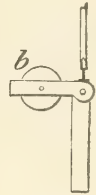


Fig. 132.



rammer *E*. This series of rammers *E* moves freely in a horizontal direction, supported on straight edges at each extremity, and is moved by the partial rotation of the wheel *C*, as shown in Fig. 122, where *f* shows a band or chain passing over its periphery, and round the guide-pulley *f*<sup>1</sup>. This chain or band *f* is attached to a series of rammers *E*, so that the pressing of the lever *c*<sup>1</sup> which is fixed on the axis of the wheel *C* imparts motion to the rammers *E* in a horizontal direction. The moulds being in the position shown in Fig. 132, the next thing to do is to bend down the lever *c*<sup>1</sup>, thereby forcing the series of rammers *E* into contact with the sliding part *b*<sup>1</sup> of each of the moulds, and thereby pushing out the candles which are received into the grooved table *F*, raised up in exact position to receive them by the action of the scroll-piece *c*<sup>2</sup> attached to the wheel *C*, and on which the grooved table *F* is supported. The candles being forced from the moulds by the rammers are immediately secured and held stationary by depressing the lever *G*, which is provided with a series of like number of small convex pieces of pewter, formed of a section of the candle-moulds, which are attached to slight springs, as seen in Fig. 123. The lever is held down by a small catch. From what has been said of the frame of moulds *B*, it is obvious that the same action of the rammers *E*, which displaces the candles, will carry down to the moulds a fresh

supply of wicks for the succeeding candles, and, at this period, while the finished candles are secured on the table *F*, the nipper *f*, shown at Fig. 129, must be reapplied, after which the finished candles are cut off, and disposed of.

The next duty of the operator is to replace the lever *c*<sup>1</sup> in the position shown at Fig. 122, which carries back the rammer *E* along with the sliding top of the moulds to their former position, and the moulds are wicked ready for a fresh supply of tallow. This series of rammers *E* is formed of separate hollow tubes, supported in the cross-piece *g g*, each of which tubes is provided with a small spring having a slight projection on its inside by means of which, when the rammers are pressed against the sliding part of the moulds, marked *b*<sup>2</sup>, the spring gives way and catches firm hold of the notched part, as shown at Fig. 127. It is thereby enabled to bring it back to its former position, where the candles are forced from the moulds as soon as the rammers are retired, and have brought back the sliding tops *b*<sup>2</sup> of the respective moulds. The springs at their extremity, which had held *b*<sup>2</sup>, are relieved or lifted up by a series of rammers or rods, which pass up the interior of the hollow rammers, as already described. This second series of rods is fixed in a similar cross-piece marked *h h* in Fig. 124, which, as soon as the rammers are retired from the moulds, is forced forward by means of the lever *H*, and thereby the caps and the whole of the moulds marked *B* are freed from any connection with the rammers *E*. At this period the moulds are passed forward to the railroad *d d*, and replaced at the position shown at *B*<sup>1</sup>, the tallow from the receiver *A* is again supplied to them, and the process already described repeated any number of times.

The continuous wick machines for moulding tallow-candles are similar in construction to those used for stearin-candles, which will be described later on.

The darkening of the flame by scales and consequent necessity of snuffing are a defect common to all tallow-candles. Tallow having a comparatively low melting point requires thick wicks. With the use of thin wicks, which bend over and are consumed by the flame, melting off on one side and consequent guttering of the candle are unavoidable.

It remains to mention the so-called plated candles, *i. e.*, tallow-candles coated with a thin layer of wax, or of a mixture of tallow and wax, or of spermaceti, or stearic acid. Usually wax with or without an addition of tallow is used. Two methods are in vogue. By the first the mould, the lower end of which has been previously closed, is filled one-fourth or one-third with melted wax, and after having closed the top, rolled upon a table. The stopper is then removed and the superfluous wax poured out. The mould, the sides of which are now covered by a coating of wax, is wicked and filled with tallow in the usual manner. Externally such candles have the appearance of wax-candles, but the wax being less fusible forms an edge on the burning wick which becomes the higher the purer the wax and the denser the layer of it. Hence, it is best to use a mixture of about equal parts of wax and tallow. The thickness of the coating, depending as well on the heat of the wax poured into the mould as on the outside temperature, varies very much. Its only purpose being, however, to remove the smeary external surface of a tallow-candle, it is best to make it as thin as possible, and, hence, the following method for which pure wax or mixed with 10 to 15 per cent. mutton suet is used, is to be preferred. Take a smooth steel cylinder fitting loosely into the candle-mould. Coat this cylinder with tallow or fat, and then dip it into melted wax, withdrawing it immediately, whereby it will be coated with a thin layer of wax. This shell, which can be readily removed from the cylinder, is placed in the mould, and the latter, after wicking, is filled with tallow in the usual manner. The warm tallow pressing the softened shell of wax against the sides of the mould secures a uniform smoothness of the exterior surface of the candle.

The whiteness of the finished tallow-candles is improved by suspending them, especially in winter, in an airy room. Candles made from unbleached or gray tallow can be bleached by means of chlorine. The candles are hung, without touching each other, in a tight wooden box, and closing the lid hermetically, chlorine is gradually introduced. The box is provided with a pane of glass, through which the process of bleaching can be observed. When sufficiently bleached, the candles are rinsed off by dipping them in cold water and hung up to dry.



*Stearin candles.*—The principal raw materials used in the manufacture of these candles are palm-oil and tallow, though in this country lard is largely used for this purpose. The researches of W. Heintz, which complete those made by Chevreul, have taught us that these fats consist of palmitic, stearic, and oleic acids, and glycerin. The acid which Chevreul designated as margaric acid has been proved to be a mixture of palmitic and stearic acids. The so-called “stearin candles” are frequently made of a mixture of stearin (*i. e.*, a mixture of palmitic and stearic acids), and soft paraffin. Candles of this description are known abroad as Apollo and Melamyl candles. The manufacture of stearin candles consists in two chief operations, viz: 1. The preparation of the fatty acids, and 2, the conversion of these acids into candles.

The preparation of the fatty acids has been already described and illustrated in Part I., Chapter II.

By careful melting experiments, Heintz has confirmed former observations, that a mixture of two solid fatty acids, which are homologous, has a lower melting point in certain proportions of mixture than each separate acid. During these melting experiments he observed also the properties of the congealed mixtures, and found that, while some are completely crystalline, others are entirely non-crystalline.

On account of the importance of the subject, which gives to the manufacturer of stearin important points in regard to the nature and value of his products, we give Heintz's observations in the following tables :—

1. *Myristic and lauric acids.*

A mixture of		Melts at Degrees F.	Congeals at Degrees F.	Manner of congealing.
Myristic acid. $C_{14}H_{27}O.HO$	Lauric acid. $C_{12}H_{23}O.HO$			
100 parts	0 parts	138.0°	—	Scaly crystalline.
90 “	10 “	125.0	117.0	Same.
80 “	20 “	121.0	112.0	Extremely fine crystalline.
70 “	30 “	116.0	102.0	Same.
60 “	40 “	109.5	102.0	Non-crystalline.
50 “	50 “	99.0	96.0	Large-foliated crystalline.
40 “	60 “	98.0	92.0	Non-crystalline.
30 “	70 “	95.0	90.0	Non-crystalline, woolly.
20 “	80 “	101.0	91.5	Same.
10 “	90 “	106.0	97.0	Needly crystalline.
0 “	100 “	110.5	—	Scaly crystalline.

2. *Palmitic and myristic acids.*

A mixture of		Melts at Degrees F.	Congeals at Degrees F.	Manner of congealing.
Palmitic acid, $C_{16}H_{31}O.HO$	Myristic acid, $C_{14}H_{27}O.HO$			
100 parts	0 parts	143.5°	—	Scaly crystalline.
95 "	5 "	142.0	136.5	Same.
90 "	10 "	140.0	132.0	Same.
80 "	20 "	136.5	128.0	Scaly and indistinctly needly.
70 "	30 "	131.0	124.0	In extremely fine needles.
60 "	40 "	125.0	121.0	Non-crystalline, rugged.
50 "	50 "	118.0	113.5	Large-foliated crystalline.
40 "	60 "	116.5	110.5	Indistinctly foliated.
35 "	65 "	115.5	110.5	Non-crystalline, opaque.
32.5 "	67.5 "	115.0	111.0	Same.
30 "	70 "	115.0	110.5	Same.
20 "	80 "	121.0	106.0	Non-crystalline.
10 "	90 "	125.0	113.5	In long needles.
0 "	100 "	129.0	—	Scaly crystalline.

3. *Stearic and palmitic acids.*

A mixture of		Melts at Degrees F.	Congeals at Degrees F.	Manner of congealing.
Stearic acid, $C_{18}H_{35}O.HO$	Palmitic acid, $C_{16}H_{31}O.HO$			
100 parts	0 parts	156.5°	—	Scaly crystalline.
90 "	10 "	153.0	144.5	Same.
80 "	20 "	149.5	140.5	Finely needly crystalline.
70 "	30 "	145.0	139.0	Same.
60 "	40 "	140.5	134.0	Non-crystalline, rugged.
50 "	50 "	134.0	131.0	Large-foliated crystalline.
40 "	60 "	133.0	130.0	Same.
35 "	65 "	132.0	130.0	Non-crystalline, wavy, lustrous.
32.5 "	67.5 "	131.0	129.0	Same.
30 "	70 "	131.0	129.0	Non-crystalline, wavy, lustreless.
20 "	80 "	135.5	129.0	Very indistinctly needly.
10 "	90 "	140.0	130.0	Beautifully needly crystalline.
0 "	100 "	143.5	—	Scaly crystalline.

4. *Stearic and myristic acids.*

A mixture of		Melts at Degrees F.	Manner of congealing.
Stearic acid, $C_{18}H_{35}O.HO$	Myristic acid, $C_{14}H_{27}O.HO$		
100 parts	0 parts	156.5°	Scaly crystalline.
90 "	10 "	153.0	Still more distinctly scaly crystalline.
80 "	20 "	149.0	Somewhat less distinctly scaly crystalline.
70 "	30 "	145.0	Still less distinctly scaly crystalline, no trace of needles.
60 "	40 "	139.5	Commencement of scaly crystallization, no trace of needles.
50 "	50 "	130.0	Non-crystalline, opaque.
40 "	60 "	123.0	Beautifully large-foliated crystalline.
30 "	70 "	119.0	Foliated crystalline.
20 "	80 "	118.0	Indistinctly crystalline.
10 "	90 "	125.0	Non-crystalline, opaque.
0 "	100 "	129.0	Scaly crystalline.

5. *Palmitic and lauric acids.*

A mixture of		Melts at Degrees F.	Manner of congealing.
Palmitic acid, $C_{16}H_{31}O.HO$	Lauric acid, $C_{12}H_{23}O.HO$		
100 parts	0 parts	143.5°	Scaly crystalline.
90 "	10 "	139.5	Distinctly scaly crystalline.
80 "	20 "	135.0	Somewhat less distinctly scaly crystalline.
70 "	30 "	130.0	Still less distinctly scaly crystalline.
60 "	40 "	124.0	Granular, indistinctly scaly crystalline.
50 "	50 "	116.5	Almost entirely non-crystalline, opaque.
40 "	60 "	104.0	Large-foliated crystalline.
30 "	70 "	101.0	Small-foliated crystalline.
20 "	80 "	99.0	Finely crystalline, indistinct.
10 "	90 "	107.0	Non-crystalline.
0 "	100 "	110.5	Scaly crystalline.

6. *Stearic and lauric acids.*

A mixture of		Melts at Degrees F.	Manner of congealing.
Stearic acid, $C_{18}H_{35}O.HO$	Lauric acid, $C_{12}H_{23}O.HO$		
100 parts	0 parts	156.5°	Scaly crystalline.
90 "	10 "	152.5	Distinctly scaly crystalline.
80 "	20 "	148.0	Same.
70 "	30 "	143.5	Distinctly granular crystalline. [zation.
60 "	40 "	138.0	Granular, commencement of scaly crystalli-
50 "	50 "	132.5	Nearly crystalline, slightly granular.
40 "	60 "	123.5	Non-crystalline, warty.
30 "	70 "	110.0	Upon the surface, areas of small shiny crys-
20 "	80 "	101.0	Non-crystalline, warty. [tals.
10 "	90 "	107.0	Non-crystalline.
0 "	100 "	110.5	Scaly crystalline.



7. *Margaric acid (from cyancetyl) and myristic acid.*

A mixture of		Melts at Degrees F.	Manner of congealing.
Margaric acid, $C_{17}H_{33}O.HO$	Myristic acid, $C_{14}H_{27}O.HO$		
100 parts	0 parts	140.0°	Scaly crystalline.
90 "	10 "	135.0	Same.
80 "	20 "	132.0	Indistinctly crystalline.
70 "	30 "	128.0	Almost entirely non-crystalline with level
60 "	40 "	123.0	Amorphous, opaque. [surface.
50 "	50 "	115.0	Same.
40 "	60 "	114.0	Somewhat granular, crystalline.
30 "	70 "	112.5	Same, with larger grains.
20 "	80 "	120.0	Same, grains very indistinct.
10 "	90 "	125.0	Opaque, in scarcely perceptible concentric
0 "	100 "	129.0	Scaly crystalline. [needles.

8. *Margaric acid (from cyancetyl) and palmitic acid.*

A mixture of		Melts at Degrees F.	Manner of congealing.
Margaric acid, $C_{17}H_{33}O.HO$	Palmitic acid, $C_{16}H_{31}O.HO$		
100 parts	0 parts	140.0°	Scaly crystalline.
90 "	10 "	138.0	Same.
80 "	20 "	136.0	Same, though somewhat flowery.
70 "	30 "	134.5	Same.
60 "	40 "	134.0	Same.
50 "	50 "	133.0	Same.
40 "	60 "	133.0	Same.
30 "	70 "	134.5	Flowery, almost in long needles.
20 "	80 "	137.5	In long needles.
10 "	90 "	141.0	Same.
0 "	100 "	143.5	Scaly crystalline.

The mixtures of the above acids congeal partly like the pure acids, which contain 80 to 90 per cent. of palmitic acid, in beautiful long needles, in the same manner as the mixture of stearic and palmitic acids, formerly called margaric acid.

9. *Stearic and margaric acids (from cyancetyl).*

A mixture of		Melts at Degrees F.	Manner of congealing.
Stearic acid, $C_{18}H_{35}O_2.HO$	Margaric acid, $C_{17}H_{33}O_2.HO$		
100 parts	0 parts	157.0°	Scaly crystalline.
90 "	10 "	153.5	Same.
80 "	20 "	150.0	Same.
70 "	30 "	148.5	Same.
60 "	40 "	144.5	Same.
50 "	50 "	143.5	Same, but more pearly.
40 "	60 "	142.0	Same.
30 "	70 "	141.5	Same.
20 "	80 "	139.5	Same.
10 "	90 "	139.0	Same.
0 "	100 "	140.0	Scaly crystalline.

The mixtures of both fatty acids melt more freely than stearic acid, but only in a moderate degree more so than margaric acid. They congeal almost like unmixed fatty acids and also behave differently from the mixtures of stearic and palmitic acids.

With a mixture of two fatty acids, the melting point of which is itself lower than that of each of the two acids, the melting point becomes still lower by adding a determined quantity of a third acid; and, what is especially remarkable, even when this third acid has a greater atomic weight and higher melting point than the first two acids.

The melting point of a mixture of 30 parts of palmitic acid and 70 parts of myristic acid is at 115.0° F., but it sinks still lower by adding to 20 parts of the mixture up to 7 parts of stearic acid.

*Palmitic, myristic, and stearic acids.*

20 parts of a mixture of 30 parts of palmitic acid and 70 parts of myristic acid.	Stearic acid.	Melts at Degrees F.	Manner of congealing.
		115.0°	Non-crystalline.
	1 part	113.0	"
	2 parts	112.0	"
	3 "	111.0	"
	4 "	111.0	"
	5 "	112.0	"
	6 "	113.5	"
	7 "	115.0	"
	8 "	116.0	"

The melting point of a mixture of 30 parts of myristic acid and 70 parts of lauric acid, which melts at  $95.0^{\circ}$  F., changes as follows by adding to 20 parts of the mixture 1 to 10 parts of palmitic acid.

*Myristic, lauric, and palmitic acids.*

20 parts of a mixture of 30 parts of myristic acid and 70 parts of lauric acid.	Palmitic acid.	Melts at Degrees F.	Manner of congealing.
		95.0°	Non-crystalline.
	1 part	93.0	"
	2 parts	91.5	"
	3 "	90.0	"
	4 "	91.0	"
	5 "	93.0	"
	6 "	94.0	"
	7 "	95.5	"
	8 "	96.8	"
	9 "	99.0	Indistinctly fine needly.
	10 "	102.0	Fine needly.

The phenomena in melting together fatty acids are similar to those in metals. By fusing together two metals the melting point is frequently considerably lowered, and by adding a third or fourth metal the melting point sinks even below the boiling point of water. Bismuth, for instance, melts at  $475^{\circ}$  F., lead at  $633^{\circ}$  F., and tin at  $455^{\circ}$  F.; an alloy of 2 parts of bismuth, 1 part of lead, and 1 part of tin, known as Rose's metal, melts at  $200.75^{\circ}$  F., and an alloy called Wood's metal, containing cadmium as a fourth metal, melts at  $154.4^{\circ}$  F.

The fatty acids obtained by any one of the methods described in Part I., Chapter II., possess nearly the same properties. They represent white, transparent, and quite coherent cakes, which are, however, not sufficiently pure for moulding candles. The edges of the cakes are often more or less colored and soft, owing to some oleic acid not having been pressed out, while the surface of the cakes is contaminated with oxide of iron and the hair of the press-bags. For purification the cakes are treated with dilute sulphuric acid and steam, as described on page 45.

The properties of stearic acid in a mixture with palmitic acid varying from those of tallow, some modifications in the manufac-



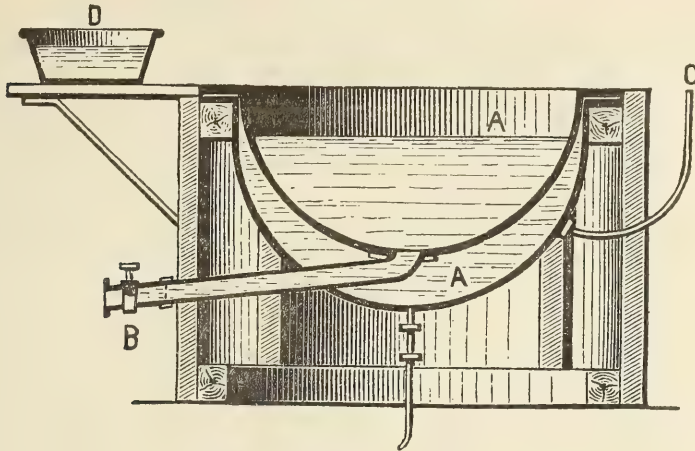
ture of candles from it are required, this being already evident from the higher melting point (about  $50^{\circ}$  to  $100^{\circ}$  F.) of the mixture of acids and its tendency of assuming a large-foliated crystalline structure on cooling, in consequence of which the candles become brittle and rough and incline towards guttering in burning. This can be overcome by the addition of up to 2 per cent. of wax or up to 20 per cent. of the cheaper paraffin. The use of arsenious acid for this purpose must be condemned as being detrimental to health, because arseniuretted hydrogen, as well as some arsenious acid, is evolved during the burning of such candles. Generally, before pouring the mass into the moulds with stirring, it is cooled almost to the congealing point, whereby it acquires a cream-like appearance and passes later on into a close-grained state. The moulds must first be heated by steam, or in an oven, as cold moulds do not completely fill up, the result being streaked candles. Although mixtures of various fatty acids have a lower melting point, as shown by Heintz's tables, they are nevertheless to be preferred, as they give harder and more transparent candles and will stand an addition of neutral fats for the cheaper manufacture of candles, as, for instance, the English composite candles (a mixture of fatty acids with coconut-oil or bleached palm-oil previously subjected to pressure).

Stearin-candles require a moderately tightly-plaited wick. In plaiting a twist is given to it which causes it to become unplaited in burning, and to twist itself out of the flame. The wick is composed of three threads (each thread having a suitable number of fine ones); one thread being shorter, and thus having a greater strain upon it than the others, gives a curvature to the whole point as soon as the melting of the candle allows it to have fair play. The wicks are soaked in one of the wick-mordants previously mentioned.

Moulding of stearin-candles by hand is only done in small factories, the first step being to melt the purified stearic-acid. To prevent it from being overheated and acquiring a brownish color, this operation is best performed in a tinned copper kettle, provided with a steam-jacket and discharge-cock, similar to the one shown in Fig. 133. The melted stearic-acid is stirred until it acquires a cream-like consistency. In the mean while the

moulder is made ready, the moulds wicked and arranged. For the more simple apparatus the same kinds of moulds with heads,

Fig. 133.

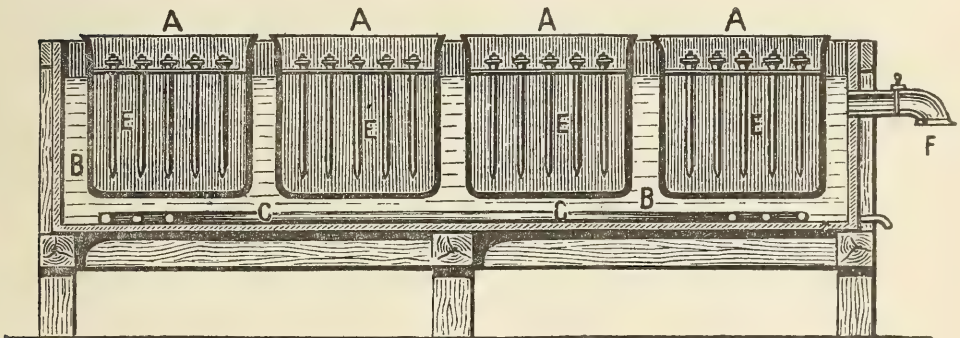


Melting-Kettle. Section.

*A*, copper kettle ; *B*, discharge-cock ; *C*, steam-pipe ; *D*, scoop.

as described for tallow-candles (Figs. 112 and 113), are used. From 20 to 30 of such moulds are suspended in a copper casing *A* (Figs. 134 and 135), which sits in the box *B*, partially filled

Fig. 134.

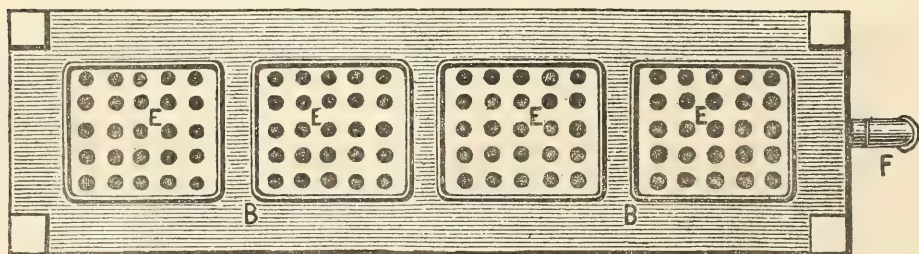


Moulding Apparatus. Longitudinal Section.

with water. The latter being heated to nearly  $212^{\circ}$  F. by means of the steam-pipe *C* lying on the bottom of *B*, the casing *A* is taken out as soon as the moulds have been heated to from  $113^{\circ}$  to  $122^{\circ}$  F., while the melting point of the fatty acid mixture lies between  $131^{\circ}$  and  $133^{\circ}$  F. The steam and air escape through the cock *F*.

The moulds, after having been heated to the above-mentioned temperature and removed from the water-box, are immediately

Fig. 135.



Moulding Apparatus. Ground Plan.

*A*, copper casing ; *B*, water-box ; *C*, steam-pipe ; *E*, moulds ; *F*, cock for escape of air and steam.

filled with the cream-like stearic-acid, and placed where they can cool. With proper precautions the candles will be of uniform appearance, and can be readily drawn from the moulds.

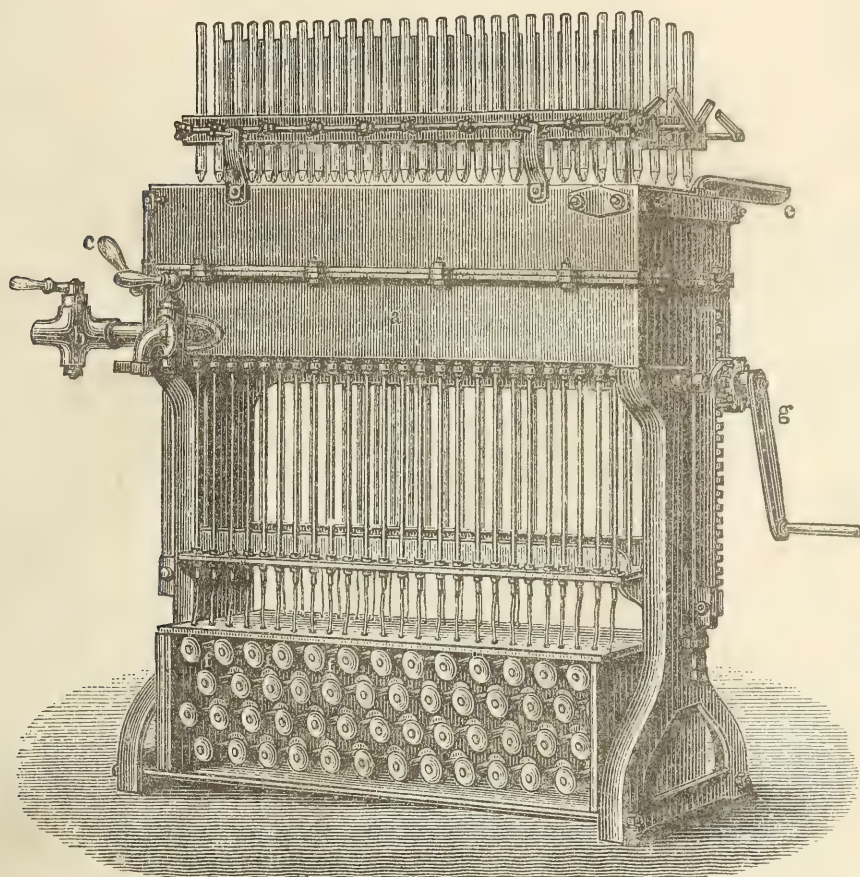
In large factories moulding machines are generally employed, so that the operation is performed uninterruptedly, the construction of these machines being such that the reeled wick is drawn through the moulds while the candles remain joined together by a short piece of wick until after the moulding is complete, the candles when cold being taken from the moulds, and the wicks cut through to separate them.

A continuous wick machine, much employed for candle-moulding, is shown in Fig. 136. The moulds, one hundred in number, are inclosed in the cast-iron box *a* ; these moulds are tubes open on each end. The tip forming the top of the candle is fastened to a tube of iron through which the wick passes. These tubes are fastened to the platform which is connected by a rack and pinion moved by the crank *g*. The wicks are reeled on bobbins inclosed in the lower case. Above the mould-case is placed an apparatus called a clamp, which grasps the finished candles as they are raised out of the moulds by the piston tubes. The clamp is made of hard wood lined with India-rubber, and acted upon by means of hinges and cranks. The mould-box is pro-



vided with suitable valves for the admission of hot or cold water, either to warm the moulds or cool them and the candles.

Fig. 136.



The method of using the machine is as follows: After having made the connection between the hot and cold water-pipes and the machine, and having connected the outlet-pipe with a drain, the machine is ready for wicking. The pistons are raised by turning the crank-handle *g* until the tips are level with the butt-ends of the candle-moulds, where they are held by a pawl catching in the pinion. A fine wire, doubled and of sufficient length to go through the tip-mould and piston, is then inserted, and extended below the piston sufficiently to enable the operator to pass the end of the wick through the loop. This permits the cotton to be drawn up through the mould; it must then be secured in any convenient manner during the first filling. The crank *g* is then returned, the melted material poured in and the

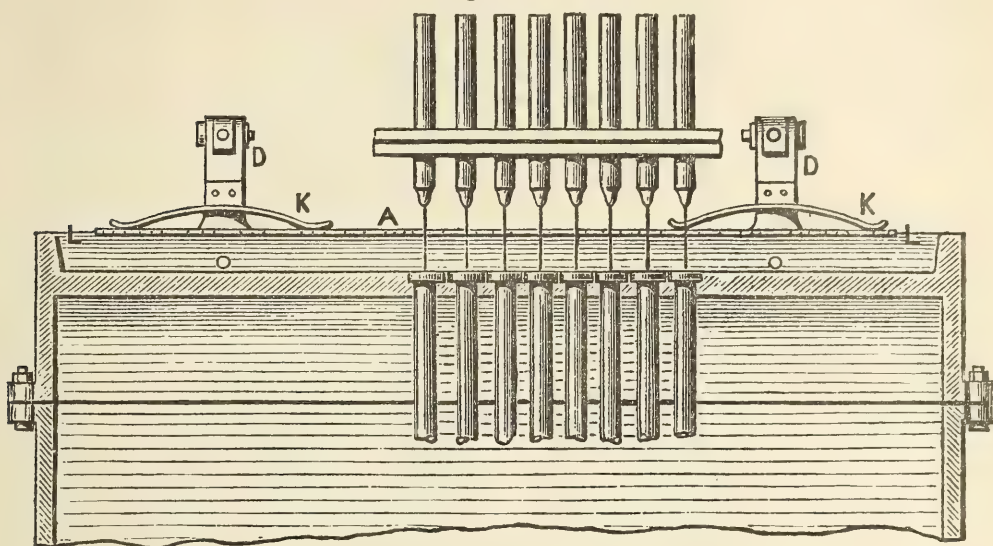
operation is complete. When nearly cold the butt ends of the candles are shaved off with a tin-scoop or a wooden-spud. The clamp should be placed open over the machine; the crank-handle *g* is then turned, and the candles are ejected into the open clamp. The latter is then closed so that each candle is held in its proper position. The crank-handle *g* is then returned to allow all the pistons to recede into their places: the wicks are thus held in a central position by the candles and the cotton-bobbins. The cotton should be slightly strained under the piston-plate. The melted material is again poured into the moulds to form a second batch; when these are nearly set the wicks are severed under the clamp, and the first batch is removed from it. The temperature of the water in the machine is easily regulated by shutting off or admitting hot or cold water as required. The internal immersion pipes, situated inside the water-box and between the rows of moulds, are perforated. These machines occupy about three feet by two feet of space, and are made to mould candles from 1 pound each to 56 to the pound. It is also possible to make candles of two different diameters, or several different lengths, in the same machine. A polished appearance is given to the candles by alternately admitting hot and cold water into the water-box. The adjustment of the temperature needs special experience, the operator's fingers forming usually his only thermometer.

The arrangement of continuous wick-machines is also explained by the following illustrations.

To attain an accurate centering of the wicks in moulding machines, Grotowski has constructed an apparatus, shown in Figs. 137, 138, 139, and 140. The apparatus consists of two side-pieces, *A* and *B*, and the middle piece *C*, which are provided with accurately-adjusted grooves 0.31 to 0.47 inch deep. These three pieces (*A*, *B*, and *C*) rest upon the upper surface *L* of the moulding machine, so that they come not in contact with the moulding material, and always remain clean. They can only be engaged, by means of the hand, by pushing the side-pieces *A* and *B* inward, and laying the middle piece *C*, which is hinged so that it can be folded together, flat. In Fig. 138, *A* is disengaged, and *B* and *C* engaged, while in the cross section, Fig. 139, all are engaged, and in the cross section 140 all disengaged.

The pieces *A* and *B* are guided by the arms *D* and *E*, which at the same time carry the candle-moulds *F*. In raising or folding

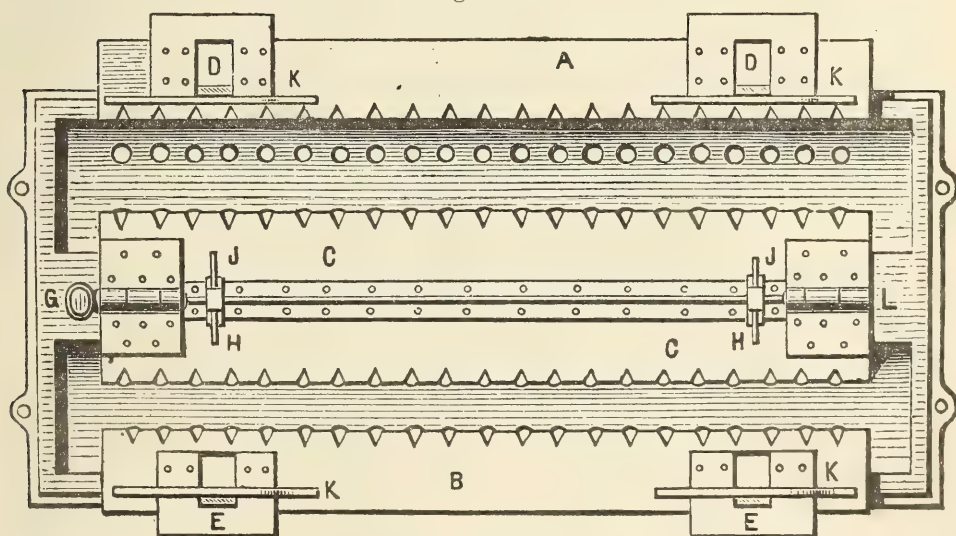
Fig. 137.



Longitudinal Section.

together the middle piece *C*, which is effected by means of the ring *G*, it is guided by the pins *H*, which also form a seat for

Fig. 138.



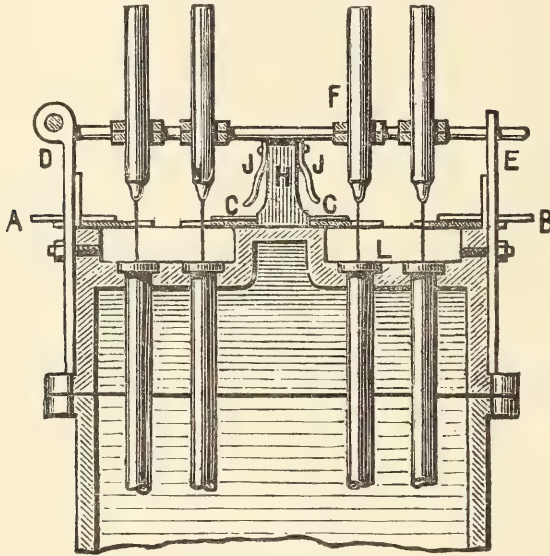
Ground Plan.

the candle-mould *F*. The springs *J* secure the middle piece *C* when raised up, while the springs *K* hold the side-pieces *A* and



*B* in every desired position. By a slight pressure upon the raised middle piece *C*, the latter slides down, and rests flat upon the upper surface *L* of the moulding machine.

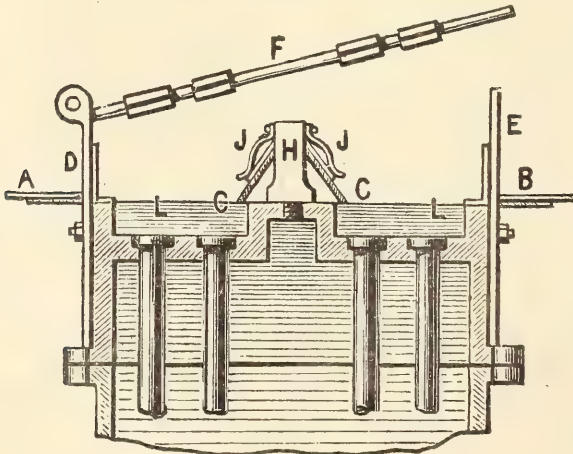
Fig. 139.



Cross Section.

When the three pieces *A*, *B*, and *C* are engaged, all the wicks are squeezed into the grooves, which are accurately adjusted to

Fig. 140.



Cross Section.

*A*, *B*, side-pieces; *C*, middle-piece; *D*, *E*, guide-arms; *F*, candle-moulds; *G*, ring; *H*, seat and guide-pins; *J*, *K*, springs; *L*, upper surface of machine.

the centres of the moulds. The melted material is then poured into the candle-mould, and, after cooling, the parts *A*, *B*, and *C*

are disengaged, in order to make the road clear for emptying the machine:

To effect an easy removal of the candles from the moulds, A. Royan has constructed a machine, shown in Figs. 141, 142, and 143, which conducts cold and warm water to the walls of the moulds, the former for the purpose of quickly cooling the material in the moulds, and the latter for the easy removal of the candles from them.

The frame of the machine carries two columns, *A*, around which are guided two plates, *B*, connected by the rod *C*. Into the plates *B* catch the ends of two racks, *D*, which are moved up and down by means of the crank *E* and the cog-wheels *F* and *G*. On the cross bars *C* are brackets, *H*, in which the ends of the wicks are secured. The moulding table contains only the copper mould-carriers *J*, in the centre of which are secured the tin moulds *K*. A trough, *L*, running the entire length of the machine, contains warm water. From this trough several tubes, *M*, enter a long pipe, *N*, containing a second pipe, provided with holes, which, by a turn of the crank *O*, can be brought opposite the branches *P*, so that, by a single turn of *O*, the supply of water can be simultaneously conducted through *P* to five moulds, which form a series, or interrupted.

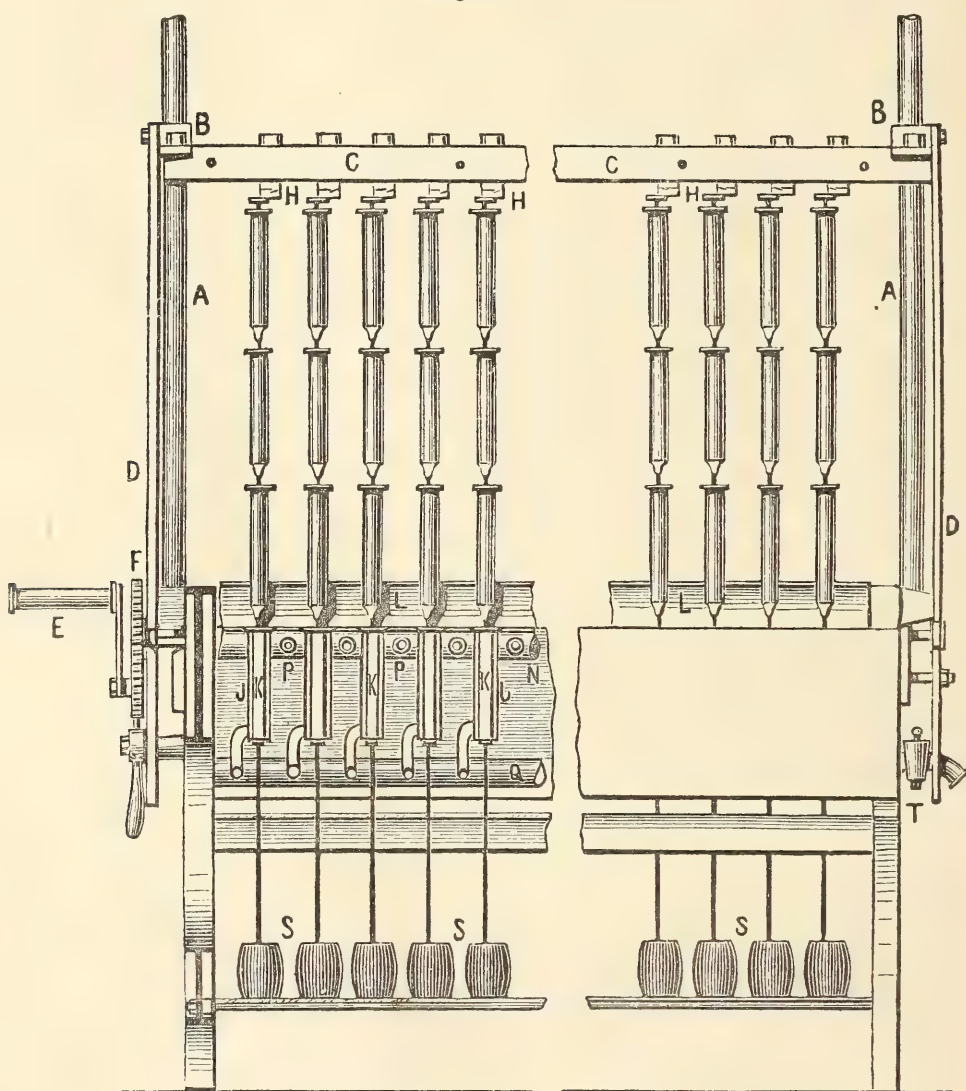
The cold water is conducted through the pipe *Q*, from which branches *R* lead to the moulds. The frame carries the bobbins *F*. After the moulds are wicked, the melted candle material is poured in and cooled by conducting cold water through the pipe *Q*. The cold water is then shut off, and after discharging that contained in the mould-carrier *J* through the cock *T*, hot water is admitted by a turn of the crank *O*, whereby the metal-moulds *K* quickly expand. If, now, by a turn of the crank *E*, the cross bars *C* with the wicks fastened to them are raised up, the candles are removed from the moulds.

Fig. 141 gives only the two ends of the machine, and shows only one-half of the longitudinal section and one-half of the longitudinal view.

The moulding machine patented by A. Motard & Co., of Berlin, is a very neat and practical arrangement, as it overcomes many of the evils of the manufacture of candles by the customary

separation of the manipulations of moulding and cutting off and regulating the weight. Some of these evils are that, in removing an excess of material, the ends of the candles frequently

Fig. 141.



Longitudinal Section and View.

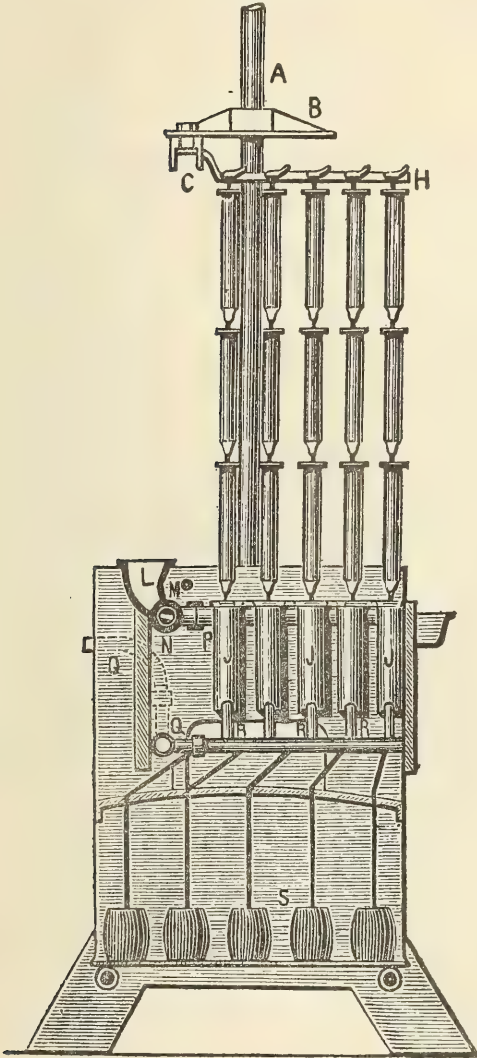
*A*, Guide columns; *B*, plates; *C*, connecting rods; *D*, racks; *E*, crank; *F*, *G*, cog-wheels; *H*, brackets; *J*, mould-carrier; *K*, tin moulds; *L*, trough for hot water; *M*, branches; *N*, long pipe for hot water; *O*, crank; *P*, branches; *Q*, cold water pipe; *R*, small pipes leading into mould-carriers; *S*, bobbins; *T*, discharge-cock for cold water.

suffer injury, and in trimming in a cutting apparatus, the candles themselves often break in consequence of the strong vibration.



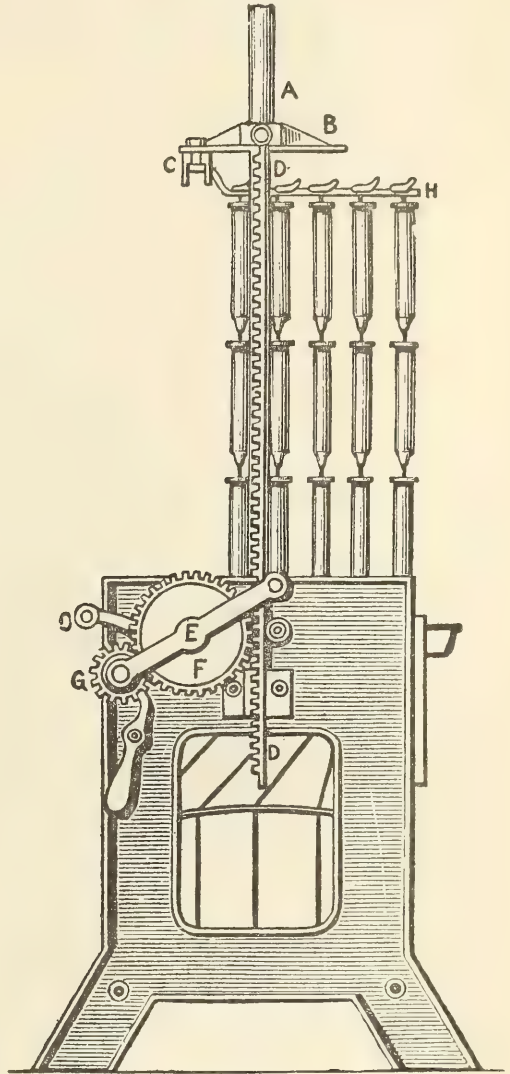
Furthermore, an exact length and weight are not always attained, and particles of the powdered candle material formed in trimming adhere, in flying around, to the candles, thus injuring their lustre.

Fig. 142.



Cross Section.

Fig. 143.

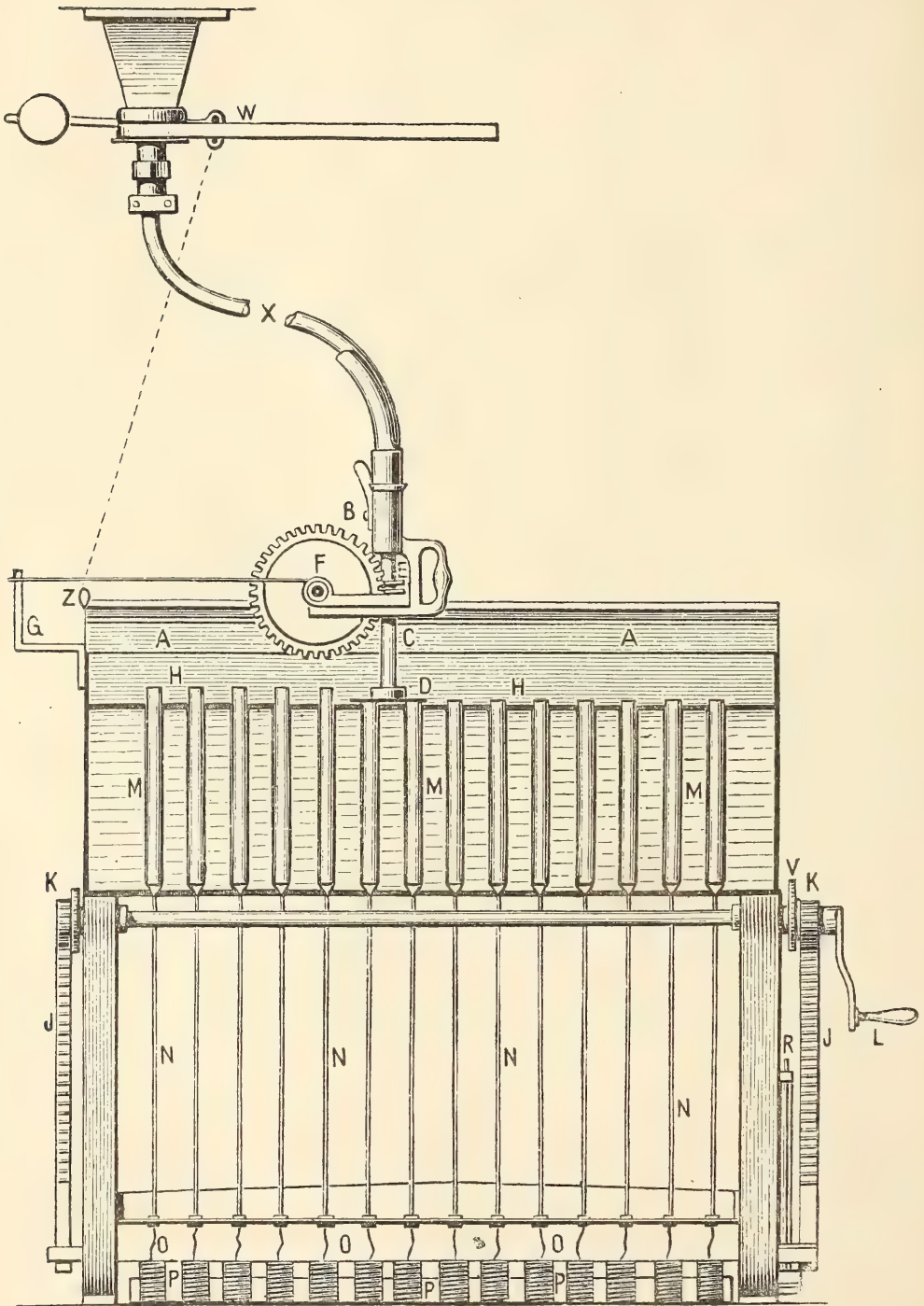


Side View.

Motard's machine, Figs. 144, 145, and 146, is a combined moulding and cutting apparatus.

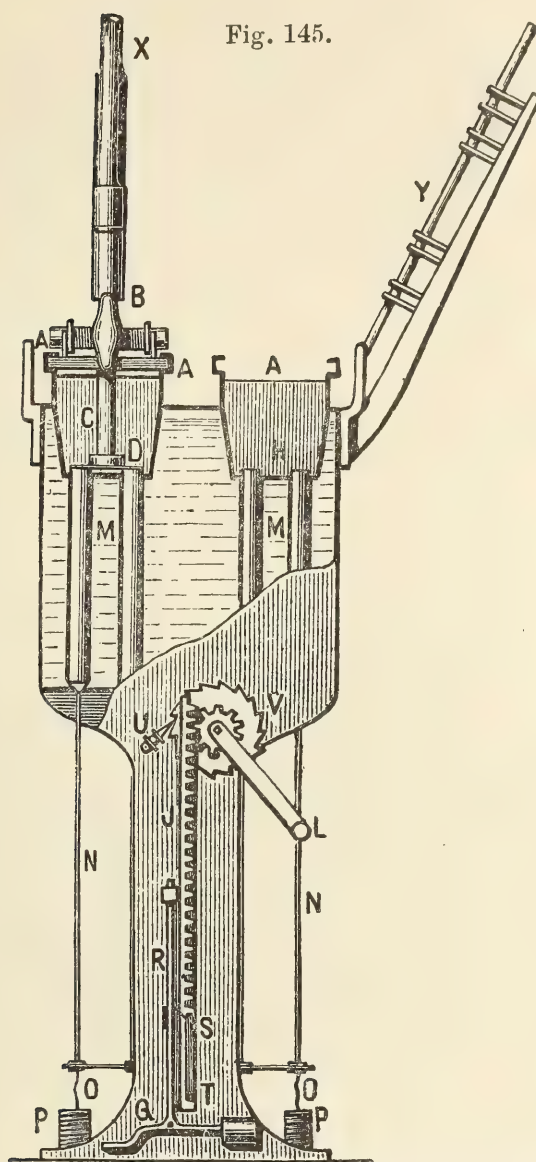
Upon the upper edge of the machine is a sliding-carriage, *A*, so secured that the cutting machine *B*, placed upon a support, can be readily engaged and disengaged. Through the support of the cutting machine passes a vertical shaft *C*, which

Fig. 144.



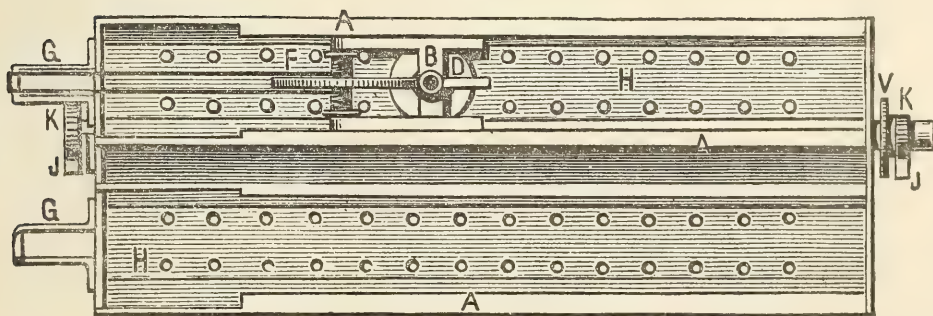
Longitudinal Section.

*A*, sliding-carriage; *B*, support of the cutting-machine; *C*, vertical shaft; *D*, circular saw; *E*, endless screw; *F*, cog-wheel; *G*, brace; *H*, moulding-box; *J*, rack; *K*, cog-wheels; *L*, crank; *M*, moulds; *N*, long tubes; *O*, wicks;



Cross-Section and Side View.

Fig. 146.



Ground-Plan.

*P*, bobbins; *Q*, treadle; *R*, rod; *S*, shiftable nose; *T*, bridge of racket; *U*, shift-able pawl; *V*, ratchet-wheel; *W*, belt-guide; *X*, flexible shaft; *Y*, clamp arrangement; *Z*, ring.



carries on the lower end a circular saw *D*, and, further up, the endless screw *E*. This screw catches into the cog-wheel *F*, upon the axis of which a cord stretched over the brace *G* winds itself up, so that the support with the revolving circular saw moves automatically in the sliding carriage from one end of the machine to the other.

The method of using the machine is as follows :—

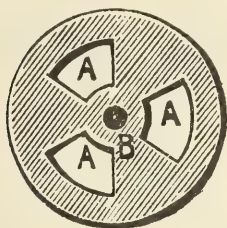
The candle material is brought into the moulding box *H*. After cooling, the ends of the candles are pushed a determined distance from the moulds by means of a contrivance consisting of the rack *J* and the cog-wheel *K*, worked by the crank *L*. The rack carries a frame with a number of long tubes, *N*, corresponding to the number of moulds, *M*. By the tubes *N* being lifted up, the candles are pushed upward, and the wick *O* is unwound from the bobbins and drawn through the tubes *N*. The treadle arrangement *Q* moves a rod *R*, which carries a shiftable nose, *S*. In lifting up the tubes the bridge *T* of the racket *J* pushes against the nose *S*, so that the candles are cut off of a determined length and weight. With the first stroke the shiftable pawl *U* falls into the ratchet-wheel *V*, thus preventing the latter from retrograding. After the first stroke the motion is transferred by the flexible shaft *X*, consisting of wire spirals, to the circular-saw *D*, and the latter, moving automatically forward, cuts off the pieces of the candles projecting over the edge of the moulds. By now moving the treadle downward, the nose *S* is withdrawn and the further removal of the candles from the moulds effected by turning the crank *L*. The candles pass into the clamp arrangement *Y*, where they are held until the tubes *N* are again lowered and the moulds have been refilled. The wicks are then cut off with a knife and the clamp *Y* turned up, so that the candles can be readily removed.

The circular saw is automatically disengaged at the end of its movement by the support *B* pushing against a ring, *Z*. This liberates the weight on the belt-guide *W*, whereby the belt is transferred from the fast to the loose pulley.

One defect of candles is that, especially when carried about, more fat is melted than is required for feeding the flame, which runs down on the candles and drops upon the floor, clothing, etc.

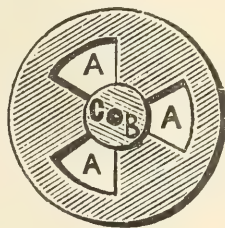
According to Urbain, this annoyance can be overcome by making the candles with any desired number of channels, as shown in Figs. 147 and 148. The excess of fat melted, it is claimed, runs into these channels, and congealing again contributes to the nourishment of the flame. These candles are made in the usual manner, but the moulds are provided with hollow, slightly conical tubes, which on filling the moulds form the channels *A*. To

Fig. 147.



Section.

Fig. 148.



Section.

*A*, channel ; *B*, wick ; *C*, inside candle.

remove the candles from the tubes, it is necessary to heat the latter by injecting a jet of steam.

Fig. 148 shows another mode of moulding. A small candle, *C*, enveloping the wick *B*, is first prepared, and the interior portion of the candle filled out with it.

The lower end of the candle is generally not consumed, and causes annoyance by dirtying the candlestick, etc. To avoid this Trenchon makes the candles of two parts, the lower one, which is about  $\frac{3}{4}$  to 1 inch long, consisting of a readily moulded material, such as plaster of Paris, etc. The latter is first poured into the mould and then the candle material. The wick unites both masses, and the candle forms a coherent body of the same shape as ordinary candles.

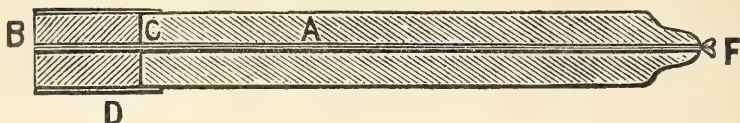
In Figs. 149 and 150 the candle material *A* and plaster of Paris *B* are separated by a straight line, *C*. The plaster of Paris can also be poured into a paper mould drawn over the candle, which is, however, not absolutely necessary. Figs. 151 and 152 show an arrangement which renders entire combustion possible. The candle material *A* catches with projections of any shape into the incombustible portion. These projections also prevent the

Fig. 149.



Cross-Section.

Fig. 150.



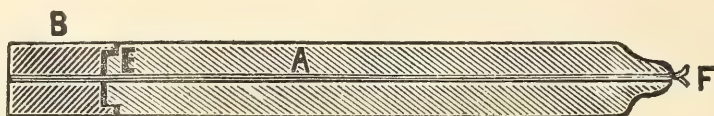
Longitudinal Section.

Fig. 151.



Cross-Section.

Fig. 152.



Longitudinal Section.

Candles with Incombustible Ends.

*A*, candle material ; *B*, incombustible mass ; *C*, division line ; *D*, paper mould ; *E*, projections into the candle material ; *F*, wick.

two portions from becoming separated by the necessary force employed in placing the candle in the candlestick.

*Spermaceti candles and paraffin candles.*—On account of their dryness and alabaster-like transparent whiteness, the better qualities of spermaceti, which are distinguished by a foliated crystalline structure, constitute a noble material for candles. Poorer qualities have a more tallowy appearance and are not entirely dry to the touch. Spermaceti-candles are made extensively, if not exclusively, in England, where they were first manufactured about 1770. These candles have always been greatly prized for their transparent whiteness, high illuminating power, and regular burning, and notwithstanding their costliness are largely used and exported to British India. In order to check the great tendency of spermaceti to crystallize, about three per cent. of the best quality of wax or of paraffin is added to the melted material. The moulding is executed in essentially the same manner as stearin candles, only the spermaceti must be so hot, about 140° F., that the portion congealing on the sides of the mould, the first moment on pouring in the mass, becomes again fluid. In cooling spermaceti contracts to such an extent that deep cavities are formed around the wick which have subsequently to be filled up.



The same trouble occurs in moulding paraffin-candles. Formerly the paraffin separated from petroleum was known as "belmontine," while the term paraffin was applied to the product obtained by the dry distillation of ozokerite, brown coal (lignite), bituminous slate, boghead coal, etc. The paraffin employed for making candles is a mixture of paraffin having different melting points.

Paraffin from boghead coal melts at from	.	.	113° to 124° F.
" " brown coal " "	.	.	129° (on an average).
" " peat " "	.	.	116°
" " Rangoon oil or tar melts at from	.	.	142°
" " ozokerite melts at from	.	.	132.5° to 179.5°

Paraffin is one of the most beautiful and pleasing materials for the manufacture of candles, but the latter have a tendency to become bent on standing for some time in a candlestick. To prevent this some stearic acid is added, the quantity depending on the melting point of the paraffin, and upon the season of the year. The quantity of stearin thus added to paraffin amounts to 3 to 15 per cent., while, as already mentioned, paraffin to the amount of 15 to 20 per cent. is added to stearin-candles, so that the material employed at the present time, for stearin- and paraffin-candles, is actually a mixture of paraffin and stearin in varying proportions.

The first paraffin-candles ever made were manufactured by Messrs. Field, of Lambeth, from paraffin extracted from Irish peat, long before paraffin was seen or known elsewhere than as small specimens in chemical laboratories.

To obtain solid candles with a hard fracture, and which in breaking crack and rattle, it is necessary to heat the paraffin to at least 140° to 158° F., and the moulds to about 113° F. The latter being filled are quickly immersed in cold water, whereby the candles suddenly solidify, and are thus prevented from becoming crystalline and opaque instead of transparent as desired.

For the black paraffin candles, sometimes used at funeral ceremonies, the paraffin is heated to nearly its boiling point together with some anacardium shells,\* the resin of which is absorbed by

\* The shells of the fruit *Semecarpus anacardium*, L., or cashew apple.

the paraffin, the latter becoming very dark-brown, and exhibiting after cooling a black color similar to that of coals. These black candles burn without smoke or smell, provided the wick be thin, which is, however, requisite in all paraffin-candles.

The various fancy patterns of candles, spiral, rope-like, etc., all require special hand-moulds.

*Manufacture of wax-candles and wax-tapers.*—Wax having the property of greatly shrinking after cooling and tightly adhering to the walls of the mould is not a very suitable material for moulding. In fact the moulding of wax-candles is now rarely, if ever, performed, but if executed is done in precisely the same manner as prescribed for stearin- and paraffin-candles. The wick for moulded wax-candles must be previously soaked with wax in order to prevent the candles from becoming, as it is technically called, honeycombed. The wax is melted on a water-bath and glass moulds are used in preference to metal ones, as well for the smooth surface glass imparts, as for the more ready removal of the candles when cold. In order to prevent the breaking of the glass-moulds they are covered with gutta-percha.

The usual manner of making wax-candles resembles somewhat the dipping of tallow-candles, only instead of the wicks being immersed, the melted mass is poured on to them. This operation is technically termed “basting.” For this purpose the wicks are suspended upon hooks in the periphery of a hoop hanging free over a furnace upon which sits a large, well-tinned copper kettle *C*, Fig. 153, containing the melted wax. The kettle is provided with a tinned sheet-iron rim *D*, and a spout *E* cut out on the side at *F*, so as to allow of a free passage of the candles suspended upon the hoop. The hoop *G*, of wood or strong sheet-iron, is provided on its outer periphery with about thirty-six iron hooks set about two inches apart. It is suspended by means of cords fastened to the periphery to a hook which is secured to a rope passing over a pulley in the ceiling, so that the hoop can be sufficiently raised to allow of the passage of the candles over the melting-kettle without touching it.

The wax is melted at a moderate heat, fresh wax being gradually added, so that during the operation a uniform temperature

is maintained by the presence of some unmelted wax in the kettle. The ladle, Fig. 154, serves for basting.

Fig. 153.

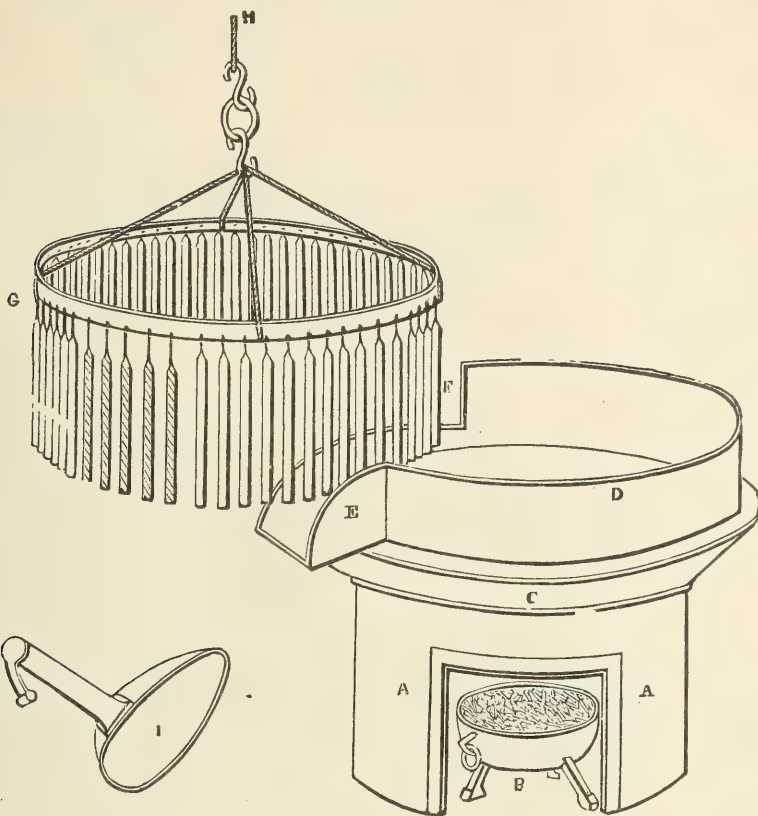


Fig. 154.

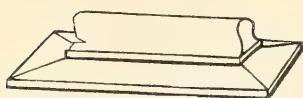
The operation of basting is executed as follows : The operator, standing by the melting kettle and having the hoop with the wicks in proper position, pours with the basting ladle first down one wick and then the next, and so on, turning the hoop at the same time and also giving a twist to the candles to insure uniform coating. When he has once gone round and the wax is sufficiently cooled, he gives the first wick a second coat, then the third, etc. until they are all of the required thickness.

The candles are then taken from the hoop, and finished and polished by rolling on a marble slab or hard-wood table with a suitable roller, the slab or table being dampened with water to prevent adhesion. The conical tops are moulded by properly shaped tools and the butt ends cut off and trimmed.



The roller (Fig. 155) for finishing and polishing the candles is made of hard wood (walnut or maple), and is about one foot long and from six to eight inches wide.

Fig. 155.



At the back, where it is about three inches thick, it is provided with a handle, and its lower surface is made as smooth as possible.

The large sized altar-candles, many of which weigh twenty to forty pounds, are sometimes made by basting, but generally by hand. The wick, consisting partially of linen and partially of cotton-yarn, is first soaked with wax, or covered with that material cut into long strips, rendered soft and kneadable by the aid of warm water, and next made up to the required thickness by rolling on more wax. Or a quantity of wax is rolled by hand into the required shape, and a longitudinal channel cut into the mass in which the wick is inserted. The channel is then filled up with wax, and the candles finished by rolling.

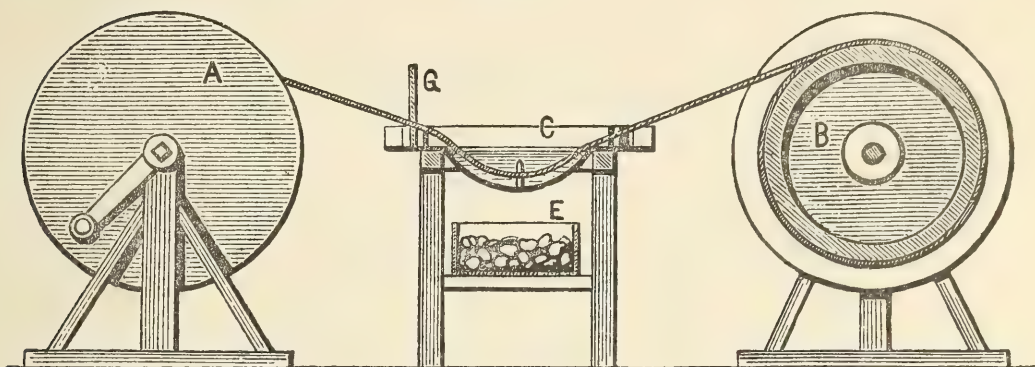
Recently Riess Brothers have constructed a press for making wax-candles. The lumps of wax to be used are placed in the press-cylinder, which is protected from cooling by the introduction of steam. The wick is inserted by being concentrically surrounded by the wax ejected from the spout of the press. A continuous candle is thus formed, which is cut up into suitable lengths.

*Wax-tapers.*—While a certain percentage of carnauba-wax, myrtle-wax, or Japan-wax is frequently added to the wax intended for candles, wax-tapers are made of wax as a base with a small percentage of tallow or fine resin and sometimes ceresin. These tapers are used for many purposes—for lighting the gas, for melting the wax for sealing letters, lighting up Christmas trees, etc.

The long, thin tapers appearing in commerce in a great variety of forms, but chiefly as a hollow coil, are made in a peculiar manner. The wick, which should be very uniform, is reeled on a drum *B*, Figs. 156, 157, 158, and 159, placed at one end of the workshop, while at the other end is a similar drum, *A*. Between these drums is placed a shallow copper-pan, *C*, filled with melted wax, which is kept warm by the charcoal fire *E*. At

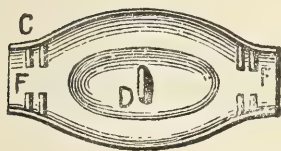
the bottom of the pan is a hook, *D*, which serves as guide for the wick. At the edge of the pan a draw-iron, *G*, is fixed, provided with circular, somewhat conical, apertures of different size, arranged in the same way as a draw-iron for wire-drawing. The

Fig. 156.



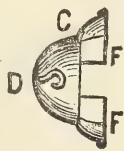
Apparatus for making Wax Tapers. Section through the Apparatus.

Fig. 157.



Ground-Plan.

Fig. 158.



Cross Section.

Fig. 159.



Draw Plate.

Copper-Pan for Melted Wax.

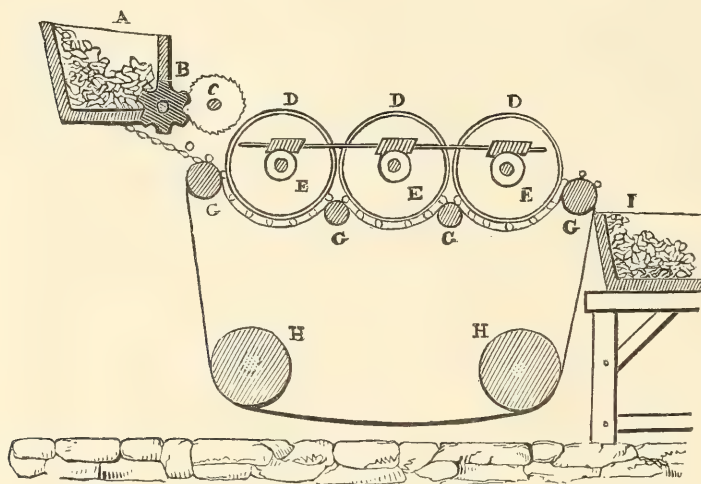
*A*, *B*, wooden drums; *C*, copper-pan; *D*, hook; *E*, charcoal basin; *F*, holder; *G*, draw-plate.

apertures can, of course, receive any other form to give other than circular-shaped tapers. The wick is drawn through the melted wax, passing under the hook *D*, and through the aperture of the drawing-iron, and next reeled on to the drum *A*, which is very slowly turned in order to give the wax time to solidify. When all the wick has been thus coated with wax, the taper, if required to be thicker, is drawn a second and even a third and fourth time through the wax and a larger sized aperture of the drawing-iron. The endless taper is then cut up into requisite lengths, which are made into a coil of a determined weight.

A good composition for wax-tapers consists of wax 8 parts, white resin 4, tallow 2, turpentine 4.

*Polishing of candles.*—Frequently the candles are polished by hand by being rubbed with a flannel-rag moistened with ammoniated alcohol. The operation is, however, effected quicker and in a more simple manner by the apparatus shown in Fig. 160, which represents a trimming and polishing machine combined.

Fig. 160.



Candle-polishing Machine.

*A*, hopper ; *B*, fluted roller ; *C*, circular saw ; *D*, cylinders covered with cloth ; *E*, pinions ; *G*, rollers ; *H*, drums ; *I*, receptacle for the polished candles.

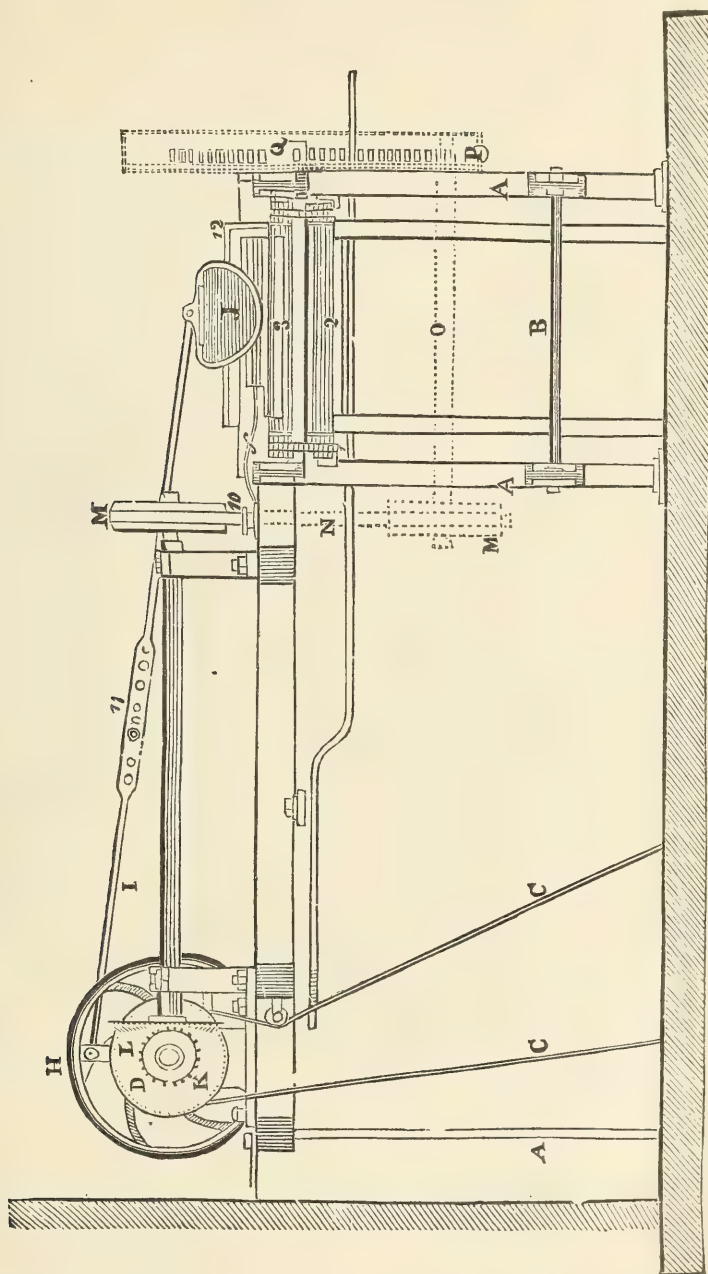
The candles are arranged in the hopper *A*, with the points towards the back of the hopper. From here they are taken singly by the fluted cylinder *B*, and carried to the circular saw *C*, which cuts the ends. They drop then upon an endless band of woollen cloth running over the rollers *G*, *G*, *G*, and around the drums *H*, *H*. Three other cylinders, *D*, *D*, *D*, covered with cloth, and run by the aid of pinions, *E*, *E*, *E*, move once around their axis in the direction opposite to that of the rollers *G*, *G*, *G*, and then in the direction of the longitudinal axis, whereby the candles, passing lengthwise, are rubbed smooth and glossy, and are finally delivered into the box *I*.

A more complete polishing machine is shown in Figs. 161 and 162. The framework *A*, *A* is strengthened by the beam *B*.



The belt *C* drives the pulley *D* or the loose pulley *E*, while the moving axle carries the two fly-wheels *H* moving the shafts *I*, *I*, and giving motion to the rubber *J* and the rubbing sheave *M*. The belt *N* connects *M* and *M*<sup>1</sup>. The latter sheave is fixed

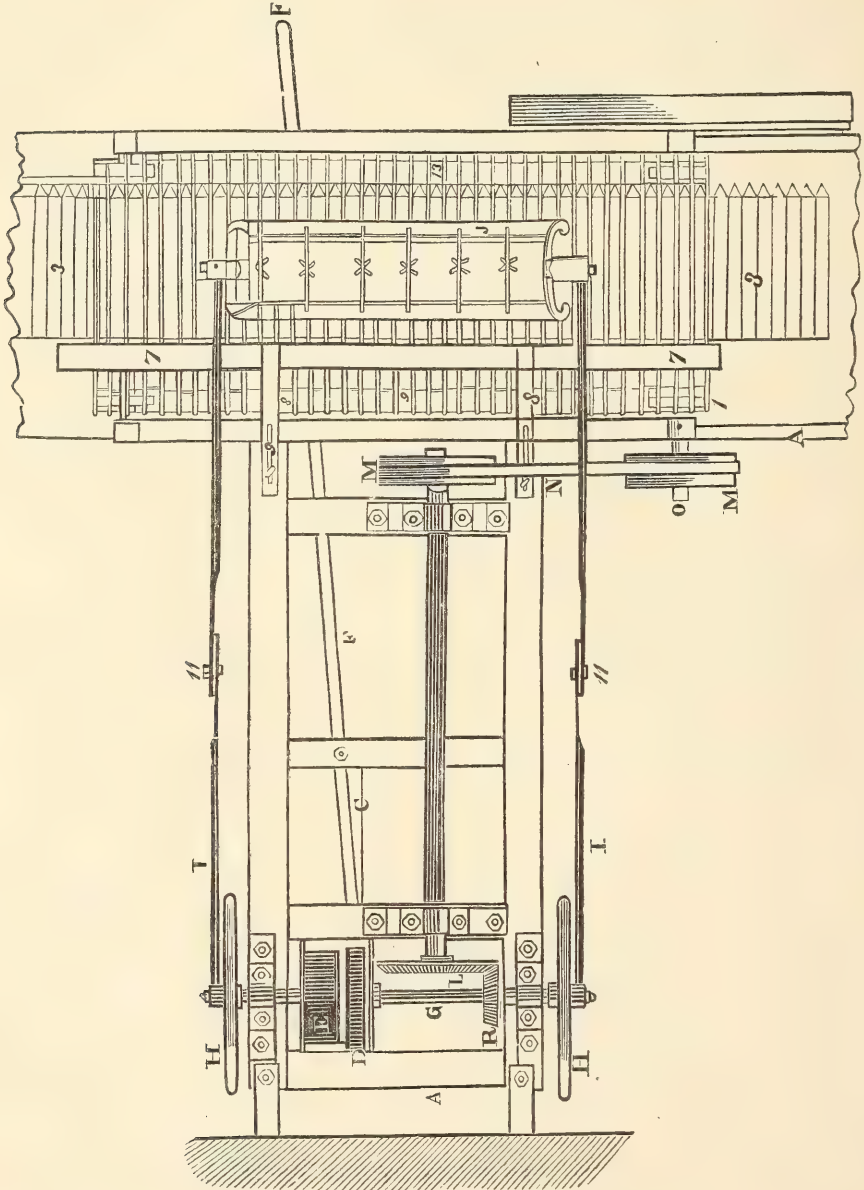
Fig. 161.



in a horizontal shaft *O*, with a pinion-wheel, *P*, at the end, working into a large cog-wheel, *Q*. The shaft *O* also carries two small wheels with square teeth, *I*, *I*, which give motion to an

endless chain, 2, composed of iron rods, and between which the candles, 3, are placed. A small table covered with woollen cloth is fixed under the endless chain on which the candles, ranging parallel with each other, roll as they are drawn forward from an

Fig. 162.



incline. The candles are retained in their position under the rubber by means of the guide 7, regulated by spring 8, 8, and, when sufficiently rubbed and polished, are deposited on the table at the other end of the machine.

It remains to be mentioned that in some factories the candles, previous to trimming and polishing, are bleached by exposure for some time to the action of air and light; but the process, being a very tedious one, it is only executed in very few cases. As a rule the candles are, however, sorted according to their degree of whiteness, some exhibiting a more yellowish tint and others a more gray-blue one. In some factories it is customary to assort the stearic acid and paraffin and prepare a special quality of candles from each kind.

*Composite candles.*—Many brands of candles under as many different names are brought into the market, which, however, vary but little in composition. The value of these candles depends upon their clean-burning and light-giving properties as well as on their handsome appearance, which in all cases should be as white and transparent as possible.

*Adamantine candles.*—These candles are made from stearic acid produced from tallow, which has a high melting point, about  $155^{\circ}$  F. They are very hard, and, though not very white or transparent, give a good white light.

*Cerophane candles.*—The mode of manufacturing the material used in the preparation of these candles is as follows: Melt together over a water-bath 100 parts of stearic acid and 10 to 11 parts of bleached beeswax, allowing the mixture to remain over the water-bath for twenty to thirty minutes without stirring or agitating. At the end of that time the fire is withdrawn, and the fluid allowed to cool until a slight film is formed on the surface. The mass is then poured direct into the moulds, previously heated to the same temperature as that of the mixture, avoiding, however, all stirring.

*Diaphane candles.*—The mass for these candles is made by melting together in a steam-jacket  $2\frac{1}{2}$  to  $17\frac{1}{2}$  pounds of vegetable wax,  $1\frac{1}{2}$  to  $10\frac{1}{2}$  pounds of pressed mutton tallow, and 22 to 46 pounds of stearic acid. The latter and the vegetable wax are the hardening ingredients. By changing the proportions between the above limits a more or less consistent mixture may be formed. The candles are moulded in the same manner as stearin candles.

*Melanyl candles* consist of equal parts of stearin and hard par-



affin. These candles combine the pleasantness of the stearin and paraffin candles without the disagreeable features of the latter.

*Parlor bougies.*—Although not bougies, a name which, properly speaking, is only applicable to wax-candles, they are very similar to them. According to Prof. Morfit, their mode of manufacture is as follows: Melt slowly over a moderate fire, in a well-tinned copper kettle, 70 pounds of pure spermaceti, and to it add piecemeal and with constant stirring 30 pounds of best white wax. By increasing the proportion of wax to 50 pounds the resulting product is much more diaphanous; however, the bougies moulded of this mixture are not as durable as candles made exclusively of wax. They are tinted in different colors, and sometimes are perfumed with essences, so that in burning they may diffuse an agreeable odor.

A still more transparent and elegant bougie is made by adding only  $6\frac{1}{2}$  pounds of wax to 100 pounds of pure, dry spermaceti.

*Transparent bougies.*—According to Debitte, the mode of manufacture is as follows: For 100 pounds of stock take 90 pounds of spermaceti, 5 pounds of purified mutton suet, and 5 pounds of wax. Melt each separately over a water-bath and to the whole, when mixed together, add 2 ounces of alum, and 2 ounces of potassium bitartrate in fine powder, and, while stirring constantly, raise the heat to  $176^{\circ}$  F. Then withdraw the fire and allow the mixture to cool to  $140^{\circ}$  F. When the impurities subside the clear liquid must be drawn off into clean pans.

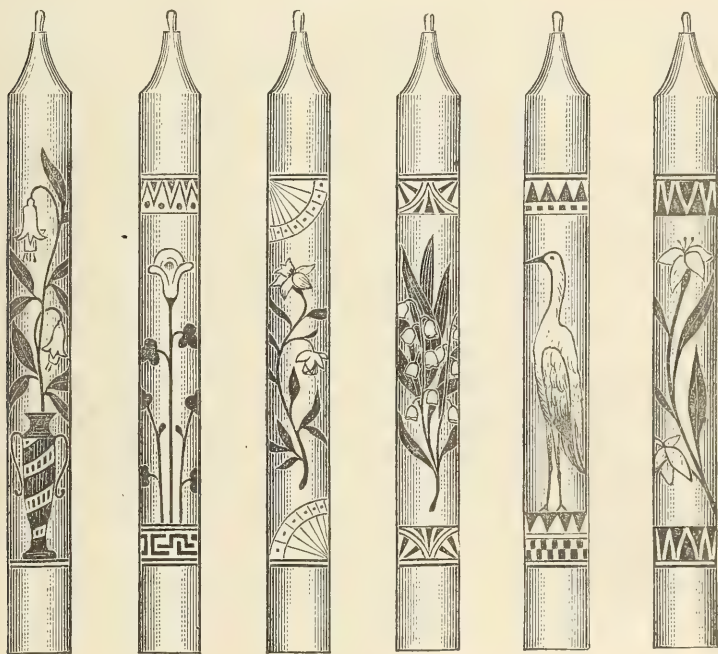
*Composition candles.*—This term is applied to candles having in their composition certain neutral fats in certain proportions, one giving to the other a quality either to improve the consistency or lighting power of the candles. The material for these candles is generally made by adding a portion of hot-pressed cocoanut stearin or palmnut stearin to stearic acid or tallow. The so-called Belmont sperm, for instance, is merely a mixture of hot-pressed stearic acid from palm and cocoanut-oils. Palm-itic acid colored by gamboge is called "Belmont-wax."

*Decorated candles.*—According to Christiani, the base of all decorated candles is a compound of stearic acid and wax or paraffin, and many that are called wax have really but little wax in their composition, nor can it be considered much to their disad-

vantage, for these composite candles, while costing one-half the price of pure wax, have nearly as good an appearance, and give as white and good a light.

As a ground for the decorations, such as shown in Fig. 163, whether done by hand or transferred by the process of decalcomanie, a suitable varnish is put on the candle. This varnish is made by dissolving gum-dammar in rectified oil of turpentine or absolute alcohol, and should be quite thick that it will not require renewing, or that one coating may be sufficient. These designs of decalcomanie are found in commerce for the purpose of the decoration of various articles, and are in nearly every conceivable design, and many are especially made for the purpose of decorating candles. It is usual for the dealers in them to give

Fig. 163.



instruction for their proper application. They are printed upon a porous paper, and when the design is pressed upon the warmed varnished surface and has hardened, the paper is moistened with water and afterwards gently rubbed off, leaving the design adhering to the candle. For decoration by hand it is customary to have the candles of the best quality, and they are prepared with a

coating of the dammar varnish above mentioned, or, still better, with a varnish made from gum-mastic.

*Coloring of candles.*—For coloring candles vegetable or aniline dyes are almost always used ; mineral colors are to be avoided, as they seriously interfere with the combustion of the candle. Grodhaus objects, as a general rule, to the coloring of tallow, with the exception of giving the candles a bluish-white tint. He claims that coloring of candles is only advisable if they are to be used shortly after being manufactured. For this he gives the following receipts :—

*For blue*, ultramarine, sulphate of copper, and aniline blue.

*For green*, distilled verdigris, Schweinfurt green, or a mixture of yellow with blue.

*For red*, alkanet root, minium, vermillion, and permanent aniline reds.

*For yellow*, annatto, gamboge, and naphthaline yellow.

*For purple or violet*, a mixture of blue with red.

By a judicious mixing of the above coloring substances, nearly all possible tints can be obtained.

The candles are sometimes only colored on the outer surface. This is done in a manner similar to that of plating candles. The moulds are filled  $\frac{1}{4}$  or  $\frac{1}{3}$  full with the colored candle material, rolled upon a table, and emptied of the liquid portion, while the solidified part adheres to the inner surface of the mould. The latter are then filled with the material for the body of the candle, care being had to have it of as low a temperature as possible, so that it will not melt the colored shell.



## APPENDIX.

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LISTS OF PATENTS RELATING TO SOAP AND CANDLES, ISSUED BY  
THE GOVERNMENT OF THE UNITED STATES OF AMERICA, FROM  
1790 TO 1888 INCLUSIVE.\*

### *Rendering of Fats.*

These patents relate chiefly to the construction of apparatus consisting of vertical tanks with flat or conical bottoms, just above which is usually a perforated diaphragm. They are heated by steam jackets, or by coils of pipe in the tank. Many of them have attachments to carry off and deodorize the resulting offensive gases, as Nos. 46,103, 46,793, 52,640, 136,827, 138,954, 248,456, and reissues 2,048 and 2,385. 259,467 is a press; 137,698 and 241,119 show compressing rollers; 39,169 has a floating skimmer; 99,252 and 188,429 perforated cylinders; 288,106 relates to fish livers; while 242,171 and 255,863 include the use of aluminium chloride.

No.	Date.	Inventor.	Residence.
2,694	June 27, 1842.	L. Montrop,	Baltimore, Md.
3,784	Oct. 9, 1844.	E. Wilson,	Cincinnati, O.
3,827	Nov. 13, 1844.	H. A. Amelung,	St. Louis, Mo.
3,844	Dec. 4, 1844.	A. Dunn,	Rotherhithe, England.
Reissue } 167 }	May 7, 1850.	E. Wilson,	Cincinnati, O.
14,397	Mar. 11, 1856.	F. Garcin,	Philadelphia, Pa.
15,942	Oct. 21, 1856.	J. J. Bates,	Brooklyn, N. Y.
18,622	Nov. 10, 1857.	A. Lapham,	"
20,856	July 13, 1858.	J. J. Bates,	"
22,152	Nov. 30, 1858.	J. L. Alberger	Buffalo, N. Y.
29,018	July 3, 1860.	Tompert and Coyle,	Louisville, Ky.
30,575	Nov. 6, 1860.	J. M. Hunter,	New York, N. Y.
32,633	June 25, 1861.	H. S. Lewis,	Chicago, Ill.
33,219	Sept. 3, 1861.	J. Turner,	"
33,969	Dec. 17, 1861.	"	"
39,169	July 7, 1863.	Peck and Glover,	New York, N. Y.
39,565	Aug. 15, 1863.	C. E. Gray,	St. Louis, Mo.
41,010	Dec. 22, 1863.	J. J. Johnston,	Allegheny City, Pa.
41,781	Mar. 1, 1864.	S. Marsh,	Chicago, Ill.

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\* The American patent system was founded by Act of April 10, 1790. After the fire, which destroyed the Patent Office in 1836, the Government advertised for the patents which had been issued prior to that time. In this way numerous copies of the earlier patents were secured. This explanation seems necessary in view of the incompleteness in the earlier dates of these lists.

No.	Date.	Inventor.	Residence.
45,960	Jan. 17, 1865.	T. Hopkins,	Cincinnati, O.
46,103	Jan. 31, 1865.	C. E. Gray,	New York, N. Y.
46,204	Feb. 7, 1865.	P. Andrew,	Cincinnati, O.
47,264	April 18, 1865.	W. Adamson,	Philadelphia, Pa.
47,788	May 23, 1865.	A. Black,	New York, N. Y.
47,793	Mar. 14, 1865.	C. E. Gray,	"
48,559	July 4, 1865.	D. H. Kaufman,	Kokams, Ind.
Reissue } 2,048 }	Aug. 8, 1865.	C. E. Gray,	New York, N. Y.
Reissue } 2,049 }	Aug. 8, 1865.	"	"
52,640	Feb. 13, 1866.	C. J. Everett,	"
57,103	Aug. 14, 1866.	Eckel & Schuyler,	"
57,104	Aug. 14, 1866.	"	"
59,260	Oct. 30, 1866.	W. H. Pinner,	"
59,312	Oct. 30, 1866.	C. J. Everett,	"
Reissue } 2,385 }	Oct. 30, 1866.	C. E. Gray,	"
59,350	Nov. 6, 1866.	W. Branigan,	Burlington, Ia.
Reissue } 2,663 }	June 25, 1867.	W. Perry,	North Bridgewater, Mass.
62,439	Feb. 26, 1867.	"	"
69,897	Oct. 15, 1867.	A. Broadnax,	New York, N. Y.
80,006	July 14, 1868.	C. C. and G. F. Pierson,	Philadelphia, Pa.
81,743	Sept. 1, 1868.	A. Broadnax,	New York, N. Y.
81,744	Sept. 1, 1868.	"	"
82,195	Sept. 15, 1868.	W. M. Bartram,	Philadelphia, Pa.
87,482	Mar. 2, 1869.	C. J. Everett,	New York, N. Y.
96,315	Nov. 2, 1869.	W. H. H. Glover,	Southold, N. Y.
98,897	Jan. 18, 1870.	L. Thorn,	New York, N. Y.
99,250	Jan. 25, 1870.	A. Smith,	Baltimore, Md.
99,252	Jan. 25, 1870.	"	"
99,896	Feb. 15, 1870.	W. J. Hooper and T. Hooper,	"
100,063	Feb. 22, 1870.	D. Pinger,	St. Joseph, Mo.
103,234	May 17, 1870.	J. W. Patterson,	New York, N. Y.
124,983	Mar. 26, 1872.	M. J. Stein,	"
Reissue } 5,195 }	Dec. 14, 1872.	A. Black,	"
136,129	Feb. 25, 1873.	A. Broadnax,	Mont Clair, N. J.
136,208	Feb. 25, 1873.	J. N. B. Bond,	New York, N. Y.
136,420	Mar. 4, 1873.	P. W. Dalton,	Jersey City, N. J.
136,827	Mar. 18, 1873.	H. S. Firman,	New York, N. Y.
137,698	April 18, 1873.	W. C. Marshall,	"
138,954	May 13, 1873.	S. Strunz,	Pittsburgh, Pa.
Reissue } 5,643 }	Nov. 4, 1873.	H. S. Firman,	New York, N. Y.
146,586	Jan. 20, 1874.	"	"
Reissue } 5,925 }	June 23, 1874.	A. Broadnax,	Mont Clair, N. J.
153,874	Aug. 4, 1874.	J. J. Willis,	New York, N. Y.
168,611	Oct. 11, 1875.	N. Caldwell,	Cincinnati, O.
171,613	Dec. 28, 1875.	H. Halvorson,	Cambridge, Mass.
171,756	Jan. 4, 1876.	P. Andrew,	Cincinnati, O.
172,942	Feb. 1, 1876.	"	"
Reissue } 7,305 }	Sept. 12, 1876.	J. L. Alberger,	Buffalo, N. Y.

No.	Date.	Inventor.	Residence.
178,269	June 6, 1876.	R. Bullymore,	Buffalo, N. Y.
179,233	June 27, 1876.	G. Upton,	Peabody, Mass.
179,883	July 18, 1876.	W. E. Andrew,	New York, N. Y.
181,695	Aug. 29, 1876.	A. S. Lyman,	"
182,084	Sept. 12, 1876.	A. L. Smith and A. Smith, Sr.	Cincinnati, O.
188,429	Mar. 13, 1877.	A. and L. Smith,	"
189,541	April 10, 1877.	H. Halvorson,	Cambridge, Mass.
194,194	Aug. 14, 1877.	A. Trotter,	Chicago, Ill.
200,252	Feb. 12, 1878.	R. Bullymore,	Buffalo, N. Y.
204,471	June 4, 1878.	T. J. Alberdingk,	Amsterdam, Holland.
222,277	Dec. 2, 1879.	A. Husbands,	St. Louis, Mo.
Reissue } 9,254 }	June 15, 1880.	H. Halvorson,	Cambridge, Mass.
229,446	June 29, 1880.	A. Miller and R. E. Gross,	New York, N. Y.
231,558	Aug. 24, 1880.	V. Gastard,	"
233,417	Oct. 19, 1880.	D. Jarves,	Detroit, Mich.
241,119	May 10, 1881.	O. F. Boomer,	Brooklyn, N. Y.
242,171	May 31, 1881.	V. R. von Offenheim and R. von Hai- dinger,	Vienna, Austria.
248,456	Oct. 18, 1881.	T. and J. Hainsworth,	Chicago, Ill.
251,088	Dec. 20, 1881.	W. Bell,	New York, N. Y.
251,105	Dec. 20, 1881.	R. Neill,	Chicago, Ill.
254,103	Feb. 28, 1882.	G. Baylor,	St. Louis, Mo.
254,279	Feb. 28, 1882.	G. Cosine,	New York, N. Y.
255,863	April 4, 1882.	R. D. Fowler and W. J. Huet,	Paris, France.
256,715	April 18, 1882.	J. K. Martin,	Chicago, Ill.
259,467	Mar. 14, 1882.	T. Bray Bowers,	Chester, Pa.
260,266	June 27, 1882.	R. Whelan,	Chicago, Ill.
261,259	July 18, 1882.	W. Schneider,	Lehrberg, Bavaria, Ger.
261,634	July 25, 1882.	F. Seltsam,	Forchheim, Bavaria, Ger.
262,706	Aug. 15, 1882.	F. Sulzberger,	New York, N. Y.
264,189	Sept. 12, 1882.	W. Mackler,	"
269,969	Jan. 2, 1883.	W. Spiegel,	Newport, Ky.
271,700	Feb. 6, 1883.	R. Gerstang,	St. Louis, Mo.
275,404	April 10, 1883.	L. M. Ohly,	New York, N. Y.
282,584	Aug. 7, 1883.	S. Stephens,	Indianapolis, Ind.
287,862	Nov. 6, 1883.	C. H. Robinson,	Chicago, Ill.
288,106	Nov. 6, 1883.	E. Paysant,	Lockeport, Nova Scotia.
291,535	Jan. 8, 1884.	H. Rall,	St. Louis, Mo.
295,184	Mar. 18, 1884.	Lissagaray and Leplay,	Paris, France.
299,821	June 3, 1884.	D. W. McElroy,	Keokuk, Iowa.
304,687	Sept. 9, 1884.	M. Ams,	New York, N. Y.
318,156	May 19, 1885.	V. D. Anderson,	Cleveland, O.
318,502	May 26, 1885.	H. Rall,	St. Louis, Mo.
318,503	May 26, 1885.	"	"
320,911	June 30, 1885.	L. J. Caldwell,	Chicago, Ill.
361,789	April 26, 1887.	Houseman and Sprowles,	Philadelphia, Pa.



*Decomposition of Fats.*

Decomposing of fats relates chiefly to the manufacture of glycerin, and especially to the refining thereof, in which the following agents are used in the patents as indicated: Aluminium, 345,174; calcium salts, 280,894; barium salts, 280,893; zinc, 267,753, 278,849, 293,344, 336,795; silicates, 371,127; tin, 272,510; alcohol, 2534; oxalic acid, 276,551; oxide of copper, 277,575; oxides of alkalies, 359,148; oxide of lead, 263,915; sodium chloride, 264,616; electricity, 284,862 and 373,691; by atomizer, 258,614; by steam, 309,515 and 316,104; by centrifuge, 251,992; forming sulpho-sebacic acid, 243,377; and making fatty acids by sulphuric acid, 290,835.

No.	Date.	Inventor.	Residence.
2,534	April 1, 1842.	J. H. Smith,	Brooklyn, N. Y.
11,766	Oct. 3, 1854.	R. A. Tilghman,	Philadelphia, Pa.
21,711	Oct. 5, 1858.	M. Work,	Cincinnati, O.
22,691	Jan. 25, 1859.	J. C. Appenzeller,	"
22,765	Jan. 25, 1859.	Wright and Fouché,	Paris, France.
28,315	May 15, 1860.	R. A. Tilghman,	Philadelphia, Pa.
76,974	April 21, 1868.	R. C. Barton,	Brooklyn, N. Y.
144,000	Oct. 28, 1873.	C. F. A. Simonin,	Philadelphia, Pa.
167,607	Sept. 14, 1875.	T. M. Fell,	Brooklyn, N. Y.
201,704	Mar. 26, 1878.	F. Sahlfeld,	New York, N. Y.
215,372	May 13, 1879.	A. Kuehne,	Jersey City, N. J.
242,272	May 31, 1881.	C. V. Clolus,	Paris, France.
242,702	June 7, 1881.	F. Seltsam,	Forcheim, Germany.
243,377	June 28, 1881.	A. M. Jacobs,	Moscow, Russia.
251,992	Jan. 3, 1881.	J. P. Battershall,	New York, N. Y.
255,504	Mar. 28, 1882.	H. Heckel,	Cincinnati, O.
255,505	Mar. 28, 1882.	"	"
256,613	April 18, 1882.	B. T. Babbitt,	New York, N. Y.
256,614	April 18, 1882.	"	"
262,913	Aug. 22, 1882.	F. Armandy,	Saint Fons, France.
263,915	Sept. 5, 1882.	J. K. Kessler,	Milwaukee, Wis.
264,616	Sept. 19, 1882.	E. L. M. Brochon,	Milan, Italy.
266,504	Oct. 24, 1882.	T. J. O. Farrel,	Dublin, Ireland.
267,753	Nov. 21, 1882.	V. Litzschmann,	New York, N. Y.
272,510	Feb. 20, 1883.	Arms and Litzellmann,	"
275,976	April 17, 1883.	B. T. Babbitt,	"
276,551	May 1, 1883.	J. P. Battershall,	"
277,575	May 15, 1883.	J. P. Kessler,	Milwaukee, Wis.
278,849	June 5, 1883.	F. Sahlfeld,	New York, N. Y.
280,893	July 10, 1883.	E. O. Baujard,	Aubervilliers, France.
280,894	July 10, 1883.	"	"
282,547	Aug. 7, 1883.	W. F. C. McCarty,	Berlin, Germany.
283,266	Aug. 14, 1883.	"	"
283,936	Aug. 28, 1883.	T. G. Walker,	Morristown, N. J.
284,862	Sept. 11, 1883.	M. H. Lackersteen,	Chicago, Ill.
286,448	Oct. 9, 1883.	O. Laist,	Cincinnati, O.
290,835	Dec. 25, 1883.	J. A. T. Bang and J. DeCastro,	Paris, France.
293,344	Feb. 2, 1884.	E. F. Michaud and E. N. Michaud,	Auberville, France.

No.	Date.	Inventor.	Residence.
306,832	Oct. 21, 1884.	F. H. Houghton,	New York, N. Y.
309,515	Dec. 23, 1884.	E. O. Baujard,	"
316,104	April 21, 1885.	B. T. Babbitt,	"
336,795	Feb. 23, 1886.	J. H. Clinton,	St. Louis, Mo.
345,174	July 6, 1886.	C. L. Porter,	"
359,148	Mar. 8, 1887.	R. Giebertmann,	Chicago, Ill.
371,127	Oct. 4, 1887.	A. Domeier and Otto Christian Hageman,	London, England.
373,691	Nov. 22, 1887.	H. F. D. Schwahn,	Kansas City, Mo.

### *Bleaching of Fats and Oils.*

Color is removed by sunlight from wax, 190,995 and 194,799; and by steam and water, 167,607; by sodium carbonate, potassium bichromate, and sulphuric acid, 167,607 and 216,558; by charcoal and bone-black, 171,328, and from spruce gum by filtration, 57,304.

No.	Date.	Inventor.	Residence.
57,304	Aug. 21, 1866.	H. B. Estey,	Houlton, Maine.
71,768	Dec. 3, 1867.	T. Leonhard,	Paterson, N. J.
136,881	Mar. 16, 1873.	D. D. Templeton,	New York, N. Y.
171,328	Dec. 21, 1875.	J. Tardos,	New Orleans, La.
189,867	April 24, 1877.	R. Macdonald,	"
190,995	May 22, 1877.	H. T. Yaryan,	Richmond, Ind.
194,799	Sept. 4, 1877.	"	"
216,558	June 17, 1878.	Jos. Davis,	Philadelphia, Pa.

### *Soap Manufacture.*

The class of soap manufacture has for its principal subject-matter apparatus consisting of boilers, usually open, and provided with agitators, whereby the contents can be vigorously stirred. The patents indicated by the following running numbers have for their subject something different from the above, as stated: Closed boilers, adapted to operate under pressure, 2, 17, 20, 39, 42, 44, 46, 50, 54, 58, 60, 72, 90; mixers, 25, 27, 41, 45, 48; crutchers, 49, 51, 64, 69, 71, 87; coolers, 23, 34, 40; molds and presses, 52, 53, 56, 59, 61, 66, 68, 73, 74, 81, 86, 87, 94, 108; apparatus for making soap powder, 75, 79; sheets of soap, 80; soap frames, 65, 98, 100; processes, 43, 54, 57, 84, 99, 106, 108; agitation by steam, 78; electrolysis, 101; compositions, 4.

No.	Date.	Inventor.	Residence.
1	July 19, 1830.	Zoll and Doyle,	Baltimore, Md.
2	1,300 Aug. 23, 1839.	A. Dunn,	Stamford Hill, Eng.
3	1,529 Mar. 31, 1840.	J. B. Doe,	London, Eng.
4	4,404 Mar. 7, 1846.	Vaughan and Ever- man,	Philadelphia, Pa.
5	5,269 Sept. 4, 1847.	T. Shugert,	Elizabeth, Pa.
6	9,008 June 8, 1852.	J. R. St. John,	New York, N. Y.
7	11,097 June 13, 1854.	T. C. Taylor,	Camden, N. J.
8	11,098 June 13, 1854.	"	"
9	15,432 July 29, 1856.	C. Morfit,	Baltimore, Md.
10	23,510 April 5, 1859.	S. Strunz,	Birmingham, Pa.

	No.	Date.	Inventor.	Residence.
11	36,693	Oct. 14, 1862.	D. B. Chapman,	Milford, Mass.
12	37,471	Jan. 30, 1863.	"	"
13	38,167	April 14, 1863.	H. Hungerford,	New York, N. Y.
14	38,354	April 28, 1863.	D. B. Chapman,	Milford, Mass.
15	44,142	Sept. 6, 1864.	G. M. Leslie,	Newburgh, N. Y.
16	45,342	Dec. 6, 1864.	J. B. Rand,	Fishersville, N. H.
17	47,385	April 25, 1865.	Bennett and Gibbs,	Buffalo, N. Y.
18	61,876	Feb. 5, 1867.	J. Ryan,	Waukegan, Ill.
19	64,099	April 23, 1867.	C. H. Hardy,	Charlestown, Mass.
20	64,517	May 7, 1867.	J. Gallipo,	Cohoes, N. Y.
21	78,182	May 26, 1868.	M. W. Brown,	New York, N. Y.
22	81,851	Sept. 1, 1868.	A. Watt,	Wadsworth, Eng.
23	93,972	Aug. 24, 1869.	S. R. Divine,	New York, N. Y.
24	95,377	Sept. 28, 1869.	W. C. Pugh,	High Point, N. C.
25	95,741	Oct. 12, 1869.	J. Stainthorp,	New York, N. Y.
26	97,692	Dec. 7, 1869.	W. P. Pugh,	High Point, N. Y.
27	102,330	April 26, 1870.	Stainthorp and Cole,	New York, N. Y.
28	104,104	June 14, 1870.	J. Blindes,	Brooklyn, N. Y.
29	104,739	June 28, 1870.	M. and F. Hyde,	Baltimore, Md.
30	104,796	June 28, 1870.	J. Treat,	New York, N. Y.
31	105,670	July 26, 1870.	L. Groux,	"
32	{ Reissue 4,107 }	Aug. 23, 1870.	G. M. Leslie,	Newburgh, N. Y.
33	108,584	Oct. 25, 1870.	W. F. George,	New York, N. Y.
34	114,063	April 25, 1871.	J. D. Sturges,	Chicago, Ill.
35	116,008	June 20, 1871.	I. D. Balch,	Cambridge, Mass.
36	119,018	Sept. 19, 1871.	Charles Elling,	Cleveland, O.
37	{ Reissue 4,729 }	Jan. 30, 1872.	James D. Sturges,	Chicago, Ill.
38	{ Reissue 4,730 }	Jan. 30, 1872.	"	"
39	123,473	Feb. 6, 1872.	E. H. Gibbs,	New York, N. Y.
40	124,457	Mar. 12, 1872.	J. D. Sturges,	Chicago, Ill.
41	125,736	April 16, 1872.	H. N. Humiston,	Troy, N. Y.
42	126,798	May 14, 1872.	R. Freeland,	Montreal, Canada.
43	127,827	June 11, 1872.	B. T. Babbitt,	New York, N. Y.
44	127,828	June 11, 1872.	"	"
45	131,283	Sept. 10, 1872.	C. Lehmann,	"
46	132,043	Oct. 8, 1872.	B. T. Babbitt,	"
47	134,415	Dec. 31, 1872.	J. Atkiss,	Brooklyn, N. Y.
48	134,706	Jan. 7, 1873.	C. J. Schultze,	Vienna, Austria.
49	138,953	May 13, 1873.	S. Strunz,	Birmingham, Pa.
50	140,701	July 8, 1873.	G. W. Hatfield,	Nashville, Tenn.
51	147,412	Feb. 10, 1874.	C. Lehmann,	New York, N. Y.
52	152,056	June 16, 1874.	G. C. Wenzel,	Newburgh, N. Y.
53	153,979	Aug. 11, 1874.	J. Oakley,	New York, N. Y.
54	158,268	Dec. 29, 1874.	R. Freeland,	Montreal, Canada.
55	158,720	Jan. 12, 1875.	C. Lehmann,	New York, N. Y.
56	163,232	May 11, 1875.	A. Neupert,	Buffalo, N. Y.
57	164,907	June 29, 1875.	C. M. Cresson,	Philadelphia, Pa.
58	169,472	Nov. 2, 1875.	M. J. Palmer,	Detroit, Mich.
59	172,032	Jan. 11, 1876.	Wm. H. King,	Philadelphia, Pa.
60	172,068	Jan. 11, 1876.	B. T. Babbitt,	New York, N. Y.
61	174,365	Mar. 7, 1876.	J. L. Jackson,	"
62	180,688	Aug. 1, 1876.	D. Whitaker,	Boston, Mass.
63	190,866	May 15, 1877.	J. M. Jaskmann,	Barre, Vt.
64	202,886	April 23, 1878.	S. Strunz,	Pittsburgh, Pa.
65	205,136	June 18, 1878.	H. Rathmann,	Buffalo, N. Y.



No.	Date.	Inventor.	Residence.
66 224,239	Feb. 3, 1880.	A. Stearns,	Brooklyn, N. Y.
67 224,514	Feb. 17, 1880.	B. H. Chadbourne,	New York, N. Y.
68 225,287	Mar. 9, 1880.	C. L. Ingalls,	Lowell, Mass.
69 228,319	June 1, 1880.	Cornwall and Corn- wall,	Louisville, Ky.
70 228,320	June 1, 1880.	"	"
71 228,321	June 1, 1880.	"	"
72 249,256	Nov. 8, 1881.	A. H. Pritchard,	Detroit, Mich.
73 249,532	Nov. 15, 1881.	O'Keefe and Robert- son,	Liverpool, Eng.
74 252,711	Jan. 24, 1882.	J. Atkiss,	Brooklyn, N. Y.
75 255,070	Mar. 14, 1882.	Wright and Bintliff,	Manchester, Eng.
76 259,842	June 20, 1882.	R. Freeland,	Boston, Mass.
77 265,095	Sept. 26, 1882.	J. J. Johnston,	Columbiana, O.
78 268,443	Dec. 5, 1882.	W. West,	Denver, Col.
79 271,281	Jan. 30, 1883.	Sherwood and Farns- worth,	Buffalo, N. Y.
80 273,946	Mar. 13, 1883.	H. Buczkowski,	Vienna, Austria.
81 276,326	April 24, 1883.	J. Atkiss,	Brooklyn, N. Y.
82 285,736	Sept. 25, 1883.	H. W. Dopp,	Buffalo, N. Y.
83 288,412	Nov. 13, 1883.	H. DeCastro,	New York, N. Y.
84 291,329	Jan. 1, 1884.	Gibbs and Otis,	Brooklyn, N. Y.
85 294,311	Feb. 26, 1884.	Dennis and Coram,	Lowell, Mass.
86 305,293	Sept. 16, 1884.	C. Collier.	Chicago, Ill.
87 313,564	Mar. 10, 1885.	C. E. Whitaker,	Hyde Park, Mass.
88 328,611	Oct. 20, 1885.	J. C. Ralston,	Toledo, O.
89 328,714	Oct. 20, 1885.	"	"
90 330,663	Nov. 17, 1885.	B. T. Babbitt,	New York, N. Y.
91 332,605	Dec. 15, 1885.	W. A. Grant,	Houston, Texas.
92 332,606	Dec. 15, 1885.	"	"
93 333,505	Jan. 5, 1886.	Dennis and Coram,	Lowell, Mass.
94 339,376	April 6, 1886.	F. C. Christy,	Chicago, Ill.
95 339,727	April 13, 1886.	E. C. Atkins,	Indianapolis, Ind.
96 339,936	April 13, 1886.	W. T. Schuberth,	Chicago, Ill.
97 340,979	May 4, 1886.	J. H. Clapp,	"
98 342,558	May 25, 1886.	B. T. Babbitt,	New York, N. Y.
99 347,027	Aug. 10, 1886.	N. J. Clute,	Chicago, Ill.
100 349,320	Sept. 21, 1886.	S. Rosenblatt,	New York, N. Y.
101 353,566	Nov. 30, 1886.	M. H. Lackersteen,	Chicago, Ill.
102 354,186	Dec. 14, 1886.	T. E. Gardner,	London, Eng.
103 356,991	Feb. 1, 1887.	A. Doll,	Cleveland, O.
104 358,117	Feb. 22, 1887.	J. Harris,	Cincinnati, O.
105 359,637	Mar. 22, 1887.	W. T. Schuberth,	Chicago, Ill.
106 362,856	May 10, 1887.	L. Riviere,	Paris, France.
107 370,330	Sept. 20, 1887.	W. A. Grant,	Houston, Texas.
108 371,093	Oct. 4, 1887.	Michael O'Hara,	Brooklyn, N. Y.

*Soap Cutting.*

This class only contains machines for cutting the soap into slabs or bars. The machines consist usually of traversing frames carrying wires and provided with carrying aprons which bring the blocks of soap into suitable position.

No.	Date.	Inventor.	Residence.
9,953	Aug. 23, 1853.	J. B. Duff,	New York, N. Y.
13,367	July 31, 1855.	A. Van Haagen,	Cincinnati, O.
13,368	July 31, 1855.	"	"

No.	Date.	Inventor.	Residence.
22,330	Dec. 14, 1858.	W. H. Manning,	Owego, N. Y.
24,593	June 28, 1859.	R. P. Thomas,	Syracuse, N. Y.
33,055	Aug. 13, 1861.	Smith and French,	North East, Pa.
39,784	Sept. 1, 1863.	Worsley and Dorsey,	Philadelphia, Pa.
40,627	Nov. 17, 1863.	J. H. Keller,	New Orleans, La.
44,806	Oct. 25, 1864.	R. Johnson,	Urbana, Md.
45,746	Jan. 3, 1865.	J. G. Perry,	South Kingston, R. I.
48,753	July 11, 1865.	D. Whitaker,	Roxbury, Mass.
53,645	April 3, 1866.	J. McRoberts,	New Orleans, La.
58,473	Oct. 2, 1866.	H. and A. Phelps,	Albany, N. Y.
63,898	April 16, 1867.	M. H. Howell,	New York, N. Y.
68,385	Sept. 3, 1867.	H. and A. Phelps,	Albany, N. Y.
70,205	Oct. 29, 1867.	C. H. Hardy,	Charlestown, Mass.
73,714	Jan. 28, 1868.	Jos. Hatfield,	New York, N. Y.
73,994	Feb. 4, 1868.	H. and A. Phelps,	Albany, N. Y.
78,127	May 19, 1868.	J. S. Pierson,	Brooklyn, N. Y.
79,102	June 23, 1868.	D. S. Brown, Jr.	New York, N. Y.
79,402	June 30, 1868.	H. Sargent,	Chelsea, Mass.
81,200	Aug. 18, 1868.	Palmer and Bush,	Brooklyn, N. Y.
82,971	Oct. 13, 1868.	F. B. McKehey,	Albany, N. Y.
95,926	Oct. 19, 1868.	Murphey and McGregor,	Detroit, Mich.
100,135	Feb. 22, 1870.	J. Gallipo,	Cohoes, N. Y.
102,907	May 10, 1870.	Beach and Beach,	Lawrence, Mass.
102,949	May 10, 1870.	C. Lehmann,	New York, N. Y.
105,458	July 19, 1870.	W. and H. N. Hum- iston,	Troy, N. Y.
118,304	Aug. 22, 1871.	J. B. Ultsch,	Cambridge, Mass.
144,481	Nov. 11, 1873.	J. Seibert,	New York, N. Y.
145,319	Dec. 9, 1873.	J. B. Ultsch,	Cambridge, Mass.
152,056	June 16, 1874.	G. C. Wenzel,	Newburgh, N. Y.
152,178	June 16, 1874.	J. Seibert,	New York, N. Y.
153,979	Aug. 11, 1874.	J. Oakley,	"
157,203	Nov. 24, 1874.	J. H. Keller,	New Orleans, La.
163,232	May 11, 1875.	A. Neupert,	Buffalo, N. Y.
164,400	June 15, 1875.	C. F. Sieber,	Harrisburg, Pa.
166,891	Aug. 17, 1875.	J. C. Ralston,	Indianapolis, Ind.
167,936	Sept. 21, 1875.	"	"
172,032	Jan. 11, 1876.	W. H. King,	Philadelphia, Pa.
174,365	Mar. 7, 1876.	J. L. Jackson,	New York, N. Y.
178,394	June 6, 1876.	D. Whitaker,	Boston, Mass.
179,435	July 4, 1876.	M. Van Beust,	New Albany, Ind.
193,223	July 17, 1877.	Chandler and Boesch,	Charleston, S. C.
227,643	May 18, 1880.	I. M. O'Donel,	Pittsburgh, Pa.
273,176	Feb. 27, 1883.	W. D. Smith,	Boston, Mass.
296,878	April 15, 1884.	F. S. Rutschman,	Philadelphia, Pa.

### *Soap.*

The class, soap, includes a great variety of compositions, the basis of which is of course the well-known compounds of alkali with fats or resins. Very many of them are liquid, and such are specified below as detergents, while the more peculiar additions or characters of some others are pointed out: Detergents, 125, 220, 222, 230, 233, 242, 244, 252, 259, 261, 262, 263, 266, 267, 269, 272, 273, 276, 281, 283, 290; abrasives, containing abrading powders as sand, etc., 110,

123, 135, 145, 164, 173, 181, 195, 231, 248, 251, 254, 265, 269, 279, 280, 286, 291, 296; cakes of soap of peculiar form, etc., 57, 113, 140, 152, 154, 185, 196, 198, 210, 212, 225, 250, 271, 274, 299; cotton-seed-oil, 165, 166, 253, 260; petroleum, 127, 129, 194, 204, 223, 241, 246, 284, 292; sulphur, 118, 243, 258; tobacco, 270; witch-hazel, 182, 245; plaster-of-paris, 91-93; seaweed, 3, 218; sawdust, 162; soap-root, 264, 275; aniline, 266; hydrogen peroxide, 300; tar, 288; castor-oil, 216; tank-water or offal, 4, 61, 297; perfumed soft soap, 255, 256; electrolysis, 298; antiseptic, 76, 122, 142, 186, 189, 240, 255; powdered soap, 150, 249; apparatus, 203; processes, 119, 204, 213, 247; granulating soda and potash together, 294, 295; sheets of soap, 221, 226; use of centrifuge in separating lye, 276.

No.	Date.	Inventor.	Residence.
1	Oct. 1, 1830.	J. Kennedy,	Baltimore, Md.
2	July 17, 1837.	D. E. Stilwell,	Utica, N. Y.
3	Aug. 26, 1845.	Solomon Guess,	Boston, Mass.
4	Mar. 7, 1846.	D. P. Albert,	Manchester, Eng.
5	July 27, 1852.	W. McCord,	New York, N. Y.
6	Jan. 2, 1855.	R. A. Tilghman,	Philadelphia, Pa.
7	Oct. 21, 1856.	A. Pfalz,	Saxonville, Mass.
8	Oct. 28, 1856.	G. C. Lawrence,	Winchester, Mass.
9	Mar. 3, 1857.	I. Roraback,	Caddo Parish, La.
10	May 12, 1857.	L. Wilman,	Worcester, Mass.
11	Mar. 16, 1858.	C. Morfit,	Baltimore, Md.
12	Mar. 30, 1858.	D. Crawford,	Toronto, Can.
13	May 17, 1859.	W. Dawes,	Washington Co., Tenn.
14	June 28, 1859.	E. P. Thomas,	Syracuse, N. Y.
15	Oct. 4, 1859.	N. Orcutt,	Binghamton, N. Y.
16	Nov. 29, 1859.	G. W. Tolburst,	Liverpool, O.
17	Jan. 3, 1860.	Edward Patrie,	Livingston, N. Y.
18	Jan. 27, 1860.	A. H. Platt,	Cincinnati, O.
19	Mar. 20, 1860.	W. N. Yost,	Yellow Springs, O.
20	Nov. 6, 1860.	M. A. Butler,	Mariana, Fla.
21	Nov. 20, 1860.	H. N. Wilbur,	Keokuk, Ia. [S. C.]
22	Jan. 1, 1861.	T. D. Mathews,	St. Peter's Parish,
23	April 9, 1861.	W. L. Dawson,	Lynchburg, Va.
24	Sept. 3, 1861.	H. Warren,	Goshen, Ind.
25	Sept. 24, 1861.	W. Dennis,	Providence, R. I.
26	Mar. 4, 1862.	M. D. Holbrook,	New York, N. Y.
27	April 15, 1862.	I. M. Harschbarger,	Brandonville, Vt.
28	May 6, 1862.	C. C. Lewis,	Whitewater, Wis.
29	Aug. 26, 1862.	A. K. Eaton,	New York, N. Y.
30	Sept. 23, 1862.	R. Grow,	Galesburg, Ill.
31	Oct. 21, 1862.	L. M. T. Riot,	Paris, France.
32	April 14, 1863.	H. Hungerford,	New York, N. Y.
33	May 26, 1863.	E. N. Lee,	Cambridge, N. Y.
34	Jan. 5, 1864.	Helen Rose,	Milford, Mass.
35	Jan. 26, 1864.	S. A. Seely,	Brooklyn, N. Y.
36	April 19, 1864.	J. B. Rand,	Fishersville, N. H.
37	May 17, 1864.	J. C. Tilton,	Pittsburgh, Pa.
38	July 12, 1864.	H. Jennings,	Boston, Mass.
39	July 12, 1864.	Shattuck and Daniels,	Chicago, Ill.
40	July 19, 1864.	G. Robbins,	Watertown, Mass.
41	Aug. 9, 1864.	D. B. Chapman,	Hopedale, Mass.



	No.	Date.	Inventor.	Residence.
42	43,800	Aug. 9, 1864.	C. E. Silsbee,	Detroit, Mich.
43	43,852	Aug. 16, 1864.	H. Huntingdon,	Chicago, Ill.
44	44,457	Sept. 27, 1864.	Reid and Rogers,	New York, N. Y.
45	45,141	Nov. 22, 1864.	Ed De Mortimer,	Cincinnati, O.
46	45,164	Nov. 22, 1864.	Moreau and Roberts,	New York, N. Y.
47	45,166	Nov. 22, 1864.	"	"
48	45,378	Dec. 6, 1864.	James E. Powell,	Troy, N. Y.
49	46,143	Jan. 31, 1865.	J. F. Rich,	Chatham Run, Pa.
50	46,817	Mar. 15, 1865.	D. E. Packer,	Reading, Conn.
51	49,561	Aug. 22, 1865.	Wm. Sheppard,	New York, N. Y.
52	49,997	Sept. 19, 1865.	R. W. Chappell,	Chicago, Ill.
53	50,180	Sept. 26, 1865.	E. Sprague,	Schenectady, N. Y.
54	50,877	Nov. 7, 1865.	F. Kunkel,	Chicago, Ill.
55	51,119	Nov. 21, 1865.	W. Nice,	Three Rivers, Mich.
56	53,847	April 10, 1866.	N. R. E. Mayer,	Chester Court House, S. C.
57	56,068	July 3, 1866.	G. H. Lincoln,	
58	56,259	July 10, 1866.	C. C. Parsons,	Boston, Mass.
59	57,663	Sept. 4, 1866.	S. J. Baker,	Wales, Ill.
60	58,302	Sept. 25, 1866.	J. T. Ryan,	Brooklyn, N. Y.
61	58,370	Oct. 2, 1866.	A. M. Bouton,	Newark, N. J.
62	58,919	Oct. 16, 1866.	L. H. Van Spanke- ren,	Muscatine, La.
63	59,724	Nov. 13, 1866.	W. B. Milne,	Chicago, Ill.
64	60,041	Nov. 27, 1866.	N. Orcutt,	Binghamton, N. Y.
65	60,328	Dec. 11, 1866.	S. J. Beeler,	Wales, Ill.
66	60,890	Jan. 1, 1867.	C. W. Hermance,	Albany, N. Y.
67	61,466	Jan. 22, 1867.	G. W. Rogers,	Lancaster, N. Y.
68	61,995	Feb. 12, 1867.	J. Brucker,	Chicago, Ill.
69	62,157	Feb. 19, 1867.	G. Rose,	Philadelphia, Pa.
70	62,820	Mar. 12, 1867.	A. A. Constantine,	New Providence, N. J.
71	63,314	Mar. 20, 1867.	D. Shattuck,	Buffalo, N. Y.
72	63,858	April 16, 1867.	M. H. Howell,	New York, N. Y.
73	64,250	April 30, 1867.	H. Pemberton,	Allegheny, Pa.
74	64,473	May 7, 1867.	S. J. Beeler,	Wales, Ill.
75	64,555	May 7, 1867.	S. B. Mougeot,	Paris, France.
76	65,186	May 28, 1867.	Eames and Seely,	New York, N. Y.
77	65,485	June 4, 1867.	Greenwood and Wilson,	Beloit, Wis.
78	66,218	July 2, 1867.	J. Chilcott,	Brooklyn, N. Y.
79	66,987	July 23, 1867.	T. W. Nichols,	Trout Creek, N. Y.
80	70,092	Oct. 22, 1867.	N. W. Hunter,	Elizabeth City, N. C.
81	75,170	Mar. 3, 1868.	J. L. Klein,	New York, N. Y.
82	76,213	Mar. 31, 1868.	G. W. Love,	Jackson Co., Mo.
83	78,214	May 26, 1868.	D. C. Lincoln,	North Vassalboro, Me.
84	78,530	June 2, 1868.	O. C. Loomis,	Ellenburg, N. Y.
85	78,840	June 9, 1868.	Slagle, Miller, and Hoy,	Washington, D. C.
86	82,459	Sept. 22, 1868.	H. W. Weedon,	High Point, N. C.
87	82,635	Sept. 29, 1868.	H. A. Pease,	Hartford, Conn.
88	83,996	Nov. 10, 1868.	"	"
89	85,623	Jan. 5, 1869.	E. P. Thomas,	San Francisco, Cal.
90	94,673	Aug. 24, 1869.	W. T. Bush,	Obion Co., Tenn.
91	92,651	July 13, 1869.	G. Sanger,	Beloit, Wis.
92	92,652	July 13, 1869.	"	"
93	92,653	July 13, 1869.	"	"

	No.	Date.	Inventor.	Residence.
94	92,654	July 13, 1869.	G. Sanger,	Beloit, Wis.
95	92,675	July 13, 1869.	"	"
96	95,312	Sept. 28, 1869.	E. R. Breed,	Farmington, Ill.
97	95,527	Oct. 5, 1869.	L. W. Smith,	Boston, Mass.
98	95,679	Oct. 12, 1869.	H. L. Gulden,	Robeson Township, Pa.
99	97,007	Nov. 16, 1869.	A. Warfield,	Alexandria, Va.
100	97,220	Nov. 23, 1869.	N. Orcutt,	Syracuse, N. Y.
101	98,179	Dec. 21, 1869.	C. P. McGimsey,	Memphis, Tenn.
102	99,468	Feb. 1, 1870.	H. A. Pease,	Hartford, Conn.
103	99,811	Feb. 15, 1870.	J. M. Austin,	York, Pa.
104	101,193	Mar. 22, 1870.	A. Warfield,	Alexandria, Va.
105	101,812	June 28, 1870.	D. D. W. Abbott,	Boston, Mass.
106	102,546	May 3, 1870.	G. W. Hull,	New York, N. Y.
107	102,906	May 10, 1870.	H. M. Baker,	Washington, D. C.
108	102,984	May 10, 1870.	T. F. Smith,	New York, N. Y.
109	104,549	June 21, 1870.	L. Brockett,	West Haven, Conn.
110	104,812	June 28, 1870.	D. D. W. Abbott,	Boston, Mass.
111	106,322	Aug. 16, 1870.	D. B. Chapman,	New London, Conn.
112	108,540	Oct. 18, 1870.	A. Warfield,	Philadelphia, Pa.
113	108,584	Oct. 25, 1870.	W. F. George,	New York, N. Y.
114	111,434	Jan. 31, 1871.	Hoagland and McDonald,	Columbus, O.
115	112,259	Feb. 28, 1871.	H. Loos,	Lawrence, Kan.
116	113,526	April 4, 1871.	E. A. Johnson,	Philadelphia, Pa.
117	114,393	May 2, 1871.	J. T. and P. S. Beyer,	Lathrop, Mo.
118	116,821	July 11, 1871.	F. M. Ellis,	Brooklyn, N. Y.
119	116,897	July 11, 1871.	D. H. Warren,	Montreal, Can.
120	117,722	Aug. 8, 1871.	Baker and Bullock,	Lima, Pa.
121	119,772	Oct. 10, 1871.	C. R. Kicherer,	Brooklyn, N. Y.
122	121,044	Nov. 21, 1871.	J. E. Dotch,	Washington, D. C.
123	121,787	Dec. 12, 1871.	Wm. Johnson,	New York, N. Y.
124	121,930	Dec. 19, 1871.	Dominick Cardullo,	Titusville, Pa.
125	122,094	Dec. 19, 1871.	J. B. Wilson,	Philadelphia, Pa.
126	122,281	Dec. 26, 1871.	J. M. Rall,	St. Louis, Mo.
127	125,265	April 12, 1872.	John Burke,	Sullivan, Ind.
128	126,375	May 7, 1872.	D. E. Breinig,	Brooklyn, N. Y.
129	126,504	May 7, 1872.	T. R. Walker,	Thymochtee, O.
130	126,509	May 7, 1872.	"	"
131	127,901	May 11, 1872.	J. Leetch,	London, Eng.
132	129,525	July 16, 1872.	Wm. T. Bush,	Union City, Tenn.
133	129,999	July 30, 1872.	G. Wheelock,	
134	132,154	Oct. 15, 1872.	Richard Guenther,	Oshkosh, Wis.
135	134,139	Dec. 24, 1872.	"	"
136	138,712	May 6, 1873.	R. P. Thomas,	San Francisco, Cal.
137	139,921	June 17, 1873.	George Sagar,	Chicago, Ill.
138	140,789	July 15, 1873.	F. M. Pleins,	Dubuque, Ia.
139	143,430	Oct. 7, 1873.	J. H. Wyle,	Philadelphia, Pa.
140	143,601	Oct. 14, 1873.	W. V. Wallace,	Boston, Mass.
141	145,065	Dec. 2, 1873.	Longley and Stickney,	Springfield, Mass.
142	147,413	Feb. 10, 1874.	H. S. Leshner,	New York, N. Y.
143	149,756	April 14, 1874.	W. Johnson,	"
144	152,697	June 30, 1874.	Semele Short,	Cincinnati, O.
145	153,123	July 14, 1874.	T. C. Smith,	Brooklyn, N. Y.
146	153,707	Aug. 4, 1874.	P. S. Devlan,	New York, N. Y.
147	159,640	Feb. 9, 1875.	B. F. Burke,	Sullivan, Ind.
148	160,726	Mar. 9, 1875.	I. N. Stern,	Keokuk, Ia.
149	162,529	April 27, 1875.	A. G. Campbell,	Paterson, N. J.

No.	Date.	Inventor.	Residence.
150 163,571	May 25, 1875.	J. P. Bryan,	Canton, O.
151 163,828	May 25, 1875.	G. C. Wenzel,	Newburgh, N. Y.
152 164,606	June 15, 1875.	S. Strunz,	Pittsburgh, Pa.
153 165,627	July 13, 1875.	"	"
154 165,628	July 13, 1875.	"	"
155 Reissue } 6,624 }	Aug. 31, 1875.	"	"
156 166,609	Aug. 10, 1875.	Hoge and Schultz,	Zanesville, O.
157 167,669	Sept. 14, 1875.	G. L. S. Jenifer,	Cincinnati, O.
158 168,223	Sept. 28, 1875.	Peter Burns,	San Francisco, Cal.
159 169,967	Nov. 16, 1875.	W. F. Darnoby,	Nashville, Tenn.
160 170,183	Nov. 23, 1875.	John W. Munger,	Portland, Me.
161 170,682	Dec. 7, 1875.	McGugin, Lybarger, and Hibbets,	Knox Co., O.
162 173,136	Feb. 8, 1876.	Porter and Robinson,	Malden, Mass.
163 173,982	Feb. 22, 1876.	J. McEvoy,	Pittsburgh, Pa.
164 175,631	April 4, 1876.	W. Thompson,	Allegheny, Pa.
165 175,976	April 11, 1876.	G. M. Harrison,	New Orleans, La.
166 176,105	April 11, 1876.	P. J. Weber,	Buffalo, N. Y.
167 176,423	April 25, 1876.	W. J. Burgess,	Cambridge, Mass.
168 177,607	May 16, 1876.	A. J. Woodworth,	Penn Township, Chester Co., Pa.
169 179,366	June 27, 1876.	T. H. Starzman,	Tiffin, O.
170 180,390	July 25, 1876.	S. Strunz,	Pittsburgh, Pa.
171 181,790	Sept. 5, 1876.	D. Love,	Yellowhead, Ill.
172 182,247	Sept. 12, 1876.	Turley and Fleming,	Council Bluffs, Ia.
173 182,261	Sept. 12, 1876.	C. D. Wooley,	Walden, N. Y.
174 183,060	Oct. 10, 1876.	Lewis and Menzies,	Philadelphia, Pa.
175 183,986	Oct. 31, 1876.	A. R. Stanton,	Utica, N. Y.
176 184,512	Nov. 21, 1876.	A. Dove,	Brookville, O.
177 184,990	Dec. 5, 1876.	D. Stanton,	Waymart, Pa.
178 184,991	Dec. 5, 1876.	"	"
179 187,245	Feb. 13, 1877.	J. W. Bartlett,	Moline, Ill.
180 189,175	April 13, 1877.	Baxter and Horrocks,	Fall River, Mass.
181 192,565	July 3, 1877.	Wm. Clark,	New York, N. Y.
182 194,021	Aug. 7, 1877.	R. E. Whittemore,	Essex, Conn.
183 195,267	Sept. 18, 1877.	Elliott and Alexander,	New York, N. Y.
184 196,402	Oct. 23, 1877.	W. V. Wallace,	Boston, Mass.
185 196,766	Nov. 6, 1877.	A. Quentin,	Milwaukee, Wis.
186 199,087	Jan. 8, 1878.	E. L. Moodie,	New York, N. Y.
187 203,090	April 30, 1878.	T. Taylor,	Washington, D. C.
188 204,116	May 21, 1878.	A. H. West,	Hamilton, N. Y.
189 204,842	June 11, 1878.	J. L. Pollock,	Hoboken, N. J.
190 210,889	Dec. 17, 1878.	E. A. Adams,	Washington, D. C.
191 219,538	Sept. 9, 1879.	A. Smith,	Mendota, Ill.
192 221,072	Oct. 28, 1879.	F. Knapp,	Braunschweig, Ger.
193 225,400	Mar. 9, 1880.	E. W. Lincoln,	Richmond, Ind.
194 232,922	Oct. 5, 1880.	Louis Bastet,	Brooklyn, N. Y.
195 234,539	Nov. 16, 1880.	G. P. Cole,	Johnstown, N. Y.
196 235,730	Dec. 21, 1880.	H. J. Borie,	San Francisco, Cal.
197 236,926	Jan. 25, 1881.	Wright and Wright,	Washington, D. C.
198 237,861	Feb. 15, 1881.	J. R. Hare,	Baltimore, Md.
199 238,445	Mar. 1, 1881.	A. C. Selby,	Maysville, Ky.
200 241,469	May 10, 1881.	James Wright,	Charleston, Ill.
201 241,902	May 24, 1881.	White and White,	Chicago, Ill.
202 242,298	May 31, 1881.	Herman Gasser,	Platteville, Wis.
203 242,398	May 31, 1881.	D. F. Trout,	Feesburg, O.
204 243,757	July 5, 1881.	Paul Casamajor,	Brooklyn, N. Y.



	No.	Date.	Inventor.	Residence.
205	243,936	July 5, 1881.	R. A. McCullough,	Milwaukee, Wis.
206	244,937	July 26, 1881.	B. G. Seebach,	Peru, Ill.
207	245,231	Aug. 2, 1881.	A. C. Selby,	Covington, Ky.
208	245,713	Aug. 16, 1881.	B. Greenfield,	Philadelphia, Pa.
209	245,955	Aug. 23, 1881.	Jaffe and Darm- staedter,	Charlottenburg, Ger.
210	246,220	Aug. 23, 1881.	Schultz and Hoge,	Zanesville, O.
211	246,958	Sept. 13, 1881.	A. Levett,	New York, N. Y.
212	246,989	Sept. 13, 1881.	B. M. Wilkerson,	Baltimore, Md.
213	248,314	Oct. 18, 1881.	C. S. Higgins,	Brooklyn, N. Y.
214	249,432	Nov. 8, 1881.	H. N. Wheeler,	Mystic River, Conn.
215	249,478	Nov. 15, 1881.	T. W. Nichols,	New York, N. Y.
216	249,506	Nov. 15, 1881.	De La Vega and D'Olivieria,	"
217	251,137	Dec. 20, 1881.	F. R. Robinson,	St. Louis, Mo.
218	254,487	Mar. 7, 1882.	Levy and Alexander,	Paris, France.
219	254,832	Mar. 14, 1882.	W. J. Menzies,	St. Helens, Eng.
220	257,381	May 2, 1882.	Preston and Whitney,	Phelps, N. Y.
221	259,268	June 6, 1882.	H. Buckkowski,	Vienna, Austria.
222	259,389	June 13, 1882.	E. Henderson,	New York, N. Y.
223	259,755	June 20, 1882.	William Green,	St. Lawrence, Kent, Eng.
224	260,168	June 27, 1882.	E. L. Couch,	San Francisco, Cal.
225	261,156	July 18, 1882.	W. J. Houston,	"
226	261,897	Aug. 1, 1882.	J. Bankmann,	Vienna, Austria.
227	261,922	Aug. 1, 1882.	F. Heiser,	Baltimore, Md.
228	262,745	Aug. 15, 1882.	S. Crump,	Mont Clair, N. J.
229	265,520	Oct. 3, 1882.	Oscar Liebreich,	Berlin, Germany.
230	266,207	Oct. 17, 1882.	J. Scharr,	Philadelphia, Pa.
231	267,176	Nov. 7, 1882.	Albert Ford,	Atlanta, Ga.
232	268,321	Nov. 28, 1882.	A. Van Haagen,	Philadelphia, Pa.
233	269,413	Dec. 19, 1882.	Henkle and Henkle,	Logan, O.
234	269,722	Dec. 26, 1882.	B. G. Seebach,	Peru, Ill.
235	269,820	Jan. 2, 1883.	J. K. Alpaugh,	Kansas City, Mo.
236	269,823	Jan. 2, 1883.	C. A. Breyer,	Philadelphia, Pa.
237	271,901	Feb. 6, 1883.	Albert Munder,	"
238	273,075	Feb. 27, 1883.	Henry Heckel,	Cincinnati, O.
239	273,235	Feb. 27, 1883.	J. J. Johnston,	Columbiana, O.
240	273,236	Feb. 27, 1883.	"	"
241	273,239	Feb. 27, 1883.	"	"
242	275,189	April 3, 1883.	M. J. Gikler,	Beloit, Wis.
243	275,212	April 3, 1883.	C. E. Hore,	Brooklyn, N. Y.
244	275,381	April 10, 1883.	J. A. Henry,	Platteville, Wis.
245	276,376	April 24, 1883.	Chas. J. Everett,	Tenafly, N. J.
246	278,409	May 29, 1883.	Davis and Davis,	Mont Calm, Texas.
247	281,089	July 10, 1883.	Oscar Liebreich,	Berlin, Germany.
248	281,807	July 24, 1883.	W. H. Townsend,	Philadelphia, Pa.
249	282,032	July 31, 1883.	J. K. Alpaugh,	Decatur, Ill.
250	282,145	July 31, 1883.	G. A. Wrisley,	Chicago, Ill.
251	282,913	Aug. 7, 1883.	Eugen Ludwig,	Hoboken, N. J.
252	283,403	Aug. 21, 1883.	Thomas A. Kasey,	Salem, Va.
253	284,313	Sept. 4, 1883.	James Longmore,	Liverpool, Eng.
254	284,718	Sept. 11, 1883.	Buckalew and Quinn,	New York, N. Y.
255	286,048	Oct. 2, 1883.	W. J. Menzies,	St. Helens, Eng.
256	286,049	Oct. 2, 1883.	"	"
257	286,568	Oct. 9, 1883.	C. Zimmerling,	Philadelphia, Pa.
258	287,642	Oct. 30, 1883.	J. J. Dillard,	Eureka Springs, Ark.

No.	Date.	Inventor.	Residence.	
259	289,621	Dec. 4, 1883.	W. E. Clarke,	St. Louis, Mo.
260	290,079	Dec. 11, 1883.	J. Longmore,	Liverpool, Eng.
261	290,754	Dec. 25, 1883.	R. F. Dickson,	Atoka, Tenn.
262	291,827	Jan. 8, 1884.	R. H. Withington,	Sacramento, Cal.
263	292,980	Feb. 5, 1884.	C. F. Broadbent,	Baltimore, Md.
264	293,372	Feb. 12, 1884.	H. P. Stultz,	Santa Fe, N. M.
265	293,998	Feb. 26, 1884.	Herrman Barlet,	Nuremberg, Ger.
266	294,727	Mar. 4, 1884.	T. Ewing,	Philadelphia, Pa.
267	295,882	Mar. 25, 1884.	F. S. Monroe,	Oakland, Cal.
268	296,452	April 8, 1884.	A. E. Rhoads,	Baraboo, Wis.
269	299,351	May 27, 1884.	A. S. Cluff,	Cincinnati, O.
270	299,584	June 3, 1884.	C. L. F. Rose,	New York, N. Y.
271	300,214	June 10, 1884.	W. H. Carslake,	Philadelphia, Pa.
272	302,732	July 29, 1884.	M. F. Jacobs,	New York, N. Y.
273	302,970	Aug. 5, 1884.	J. B. Zieback,	Pottsville, Pa.
274	304,913	Sept. 9, 1884.	J. M. Craig,	Brooklyn, N. Y.
275	305,272	Sept. 16, 1884.	W. H. Bicksler,	Big Spring, Texas.
276	308,968	Dec. 9, 1884.	Oscar Liebreich,	Berlin, Germany.
277	310,829	Jan. 13, 1885.	J. J. Hymer,	Louisville, Ky.
278	311,555	Feb. 3, 1885.	Henry Zahn,	Chicago, Ill.
279	314,256	Mar. 24, 1885.	O. H. Mellum,	"
280	314,447	Mar. 24, 1885.	Hoagland and Zipfel,	Toledo, O.
281	316,154	April 21, 1885.	Louis Kuhlmann,	Brooklyn, N. Y.
282	319,097	June 2, 1885.	Anna Kendall,	St. Louis, Mo.
283	319,323	June 2, 1885.	C. Robinson,	Hornellsville, N. Y.
284	319,854	June 9, 1885.	Eugen Schaal,	Stuttgart, Germany.
285	320,772	June 23, 1885.	Tardy W. Ford,	Belpre, O.
286	327,426	Sept. 29, 1885.	O. W. Young,	Chicago, Ill.
287	328,589	Oct. 20, 1885.	J. Kotrba,	St. Louis, Mo.
288	331,190	Nov. 24, 1885.	Herman Endemann,	Brooklyn, N. Y.
289	331,810	Dec. 8, 1885.	C. T. Mutchler,	Ottumwa, Ia.
290	337,035	Mar. 2, 1886.	C. H. Walker,	Sandy Lake, Pa.
291	339,426	April 6, 1886.	Jacobs and Horne,	Salt Lake City, Utah.
292	340,451	April 20, 1886.	M. S. Moot,	New York, N. Y.
293	340,565	April 27, 1886.	J. Ericksen,	Albert Lea, Minn.
294	345,714	July 20, 1886.	W. J. Menzies,	St. Helens, Eng.
295	345,715	July 20, 1886.	"	"
296	346,461	Aug. 3, 1886.	C. F. Broadbent,	Baltimore, Md.
297	347,027	Aug. 10, 1886.	N. J. Clute,	Chicago, Ill.
298	353,566	Nov. 30, 1886.	M. H. Lackersteen,	"
299	354,186	Dec. 14, 1886.	T. E. Gardner,	London, Eng.
300	355,523	Jan. 4, 1887.	F. J. Harrison,	Notting Hill, Eng.
301	359,090	Mar. 8, 1887.	M. E. Minnick,	Galveston, Texas.
302	362,942	May 17, 1887.	W. J. Dane,	Detroit, Mich.
303	370,551	Sept. 27, 1887.	Eugene McCarthy,	Sacramento, Cal.
304	371,093	Oct. 4, 1887.	Michael O'Hara,	Brooklyn, N. Y.
305	381,444	April 17, 1888.	C. Toppan,	Salem, Mass.
306	381,503	April 17, 1888.	R. A. McCullough,	Milwaukee, Wis.

*Detergents.*

Detergents are usually liquid soaps with some arbitrary addition, as borax or corn meal. The following numbers containing usually a dilute acid are adapted for cleaning metal, stone, etc.: 2, 4, 7, 8, 9, 24, 27. The following are dry powders: 11, 13, 16, 22, 26. Bran-water is found in No. 14; hydrogen peroxide ( $H_2O_2$ ) in No. 28, and No. 33 is a compound of sawdust and soap.

No.	Date.	Inventor.	Residence.
1 50,180	Sept. 26, 1865.	E. Sprague,	Schenectady, N. Y.
2 141,156	July 22, 1873.	Mark McGlenn,	Aurora, Ill.
3 141,364	July 29, 1873.	Nathan W. Lane,	Mendocino, Cal.
4 145,971	Dec. 30, 1873.	J. Sawyer,	Northfield, Va.
5 158,163	Dec. 29, 1874.	J. Braddock,	Huntington, Ind.
6 166,948	Aug. 24, 1875.	Adolph Thode,	Brooklyn, N. Y.
7 167,192	Aug. 31, 1875.	G. M. Norwood,	Beverly, Mass.
8 171,035	Dec. 14, 1875.	A. Monnier,	Sacramento, Cal.
9 174,355	Mar. 7, 1876.	M. N. Cross,	San Francisco, Cal.
10 179,366	June 27, 1876.	Thomas H. Startzman,	Tiffin, O.
11 188,549	Mar. 20, 1877.	R. Sommers,	Glastonbury, Conn.
12 189,739	April 17, 1877.	R. B. Hurd,	Paterson, N. J.
13 194,990	Sept. 11, 1877.	G. P. Cole,	Johnstown, N. Y.
14 203,712	May 14, 1878.	John Daymon,	Eddystone, Pa.
15 210,208	Nov. 26, 1878.	C. C. Parsons,	New York, N. Y.
16 216,805	June 24, 1879.	H. A. S. Park,	Indianapolis, Ind.
17 217,410	July 8, 1879.	H. G. Robertson,	Asheville, N. C.
18 218,676	Aug. 19, 1879.	Charles Kahn, Jr.,	Cincinnati, O.
19 224,841	Feb. 24, 1880.	W. H. Nordaby,	Germantown, N. Y.
20 226,920	April 27, 1880.	Edward Lyon,	New York, N. Y.
21 241,469	May 10, 1881.	James Wright,	Charleston, Ill.
22 267,455	Nov. 14, 1882.	C. C. Parsons,	Brooklyn, N. Y.
23 278,176	May 22, 1883.	Wm. H. Payne,	Westernville, N. Y.
24 309,622	Dec. 23, 1884.	H. C. Herrick,	Monroe, Wis.
25 345,715	July 20, 1886.	W. J. Menzies,	St. Helens, Eng.
26 348,179	Aug. 24, 1886.	Charles Zimmerling,	Philadelphia, Pa.
27 352,438	Nov. 9, 1886.	H. A. Arndt,	"
28 355,523	Jan. 4, 1887.	F. J. Harrison,	Notting Hill, Eng.
29 359,090	Mar. 8, 1887.	M. E. Minnick,	Galveston, Tex.
30 360,948	April 12, 1887.	Peter Brentini,	London, Eng.
31 363,764	May 24, 1887.	W. A. Putnam,	Palatine, Ill.
32 370,551	Sept. 27, 1887.	Eugene McCarthy,	Sacramento, Cal.
33 377,135	Jan. 31, 1888.	F. C. Bauer,	Philadelphia, Pa.



*Candles and Apparatus.*

This class does not include the making of wicks or the preparation of the fat before moulding. Most of the very early patents are for dipped candles, as also Nos. 85 and 100. However the larger part of this class are moulding machines. The following contain some peculiarity in the candle itself, in shape or construction: Nos. 44, 50, 52, 56, 79, 88, 92, 93, 97, 98, 101, 107, 110, 111, 117, 120, 122, 124, 129, 130, 133, 134, 135. The following relate to the composition employed: Nos. 26, 77, 78, 104, 106, 108, 112. Nos. 99 and 102 are wickless candles; Nos. 54, 80, 83, and 84 paraffin candles, and Nos. 105, 123, 125, and 131 are polishing machines; while No. 89 is a machine for ornamenting candles.

No.	Date.	Inventor.	Residence.
1	Aug. 6, 1790.	J. S. Sampson,	
2	June 2, 1794.	R. Robotham,	
3	Jan. 20, 1797.	J. S. Sampson,	
4	Mar. 22, 1798.	S. Bladenburg,	
5	Jan. 6, 1809.	S. Stansbury,	New York, N. Y.
6	Jan. 6, 1809.	W. Miller,	"
7	April 2, 1812.	J. Zwisler,	Baltimore, Md.
8	July 1, 1814.	"	New York, N. Y.
9	Aug. 1, 1815.	D. M. Randolph,	Richmond, Va.
10	Jan. 14, 1820.	W. W. Swain,	New Bedford, Mass.
11	Mar. 20, 1821.	J. Aborn,	Burlington Co., N. J.
12	Sept. 29, 1821.	F. Fuller,	New York, N. Y.
13	Dec. 4, 1821.	J. M. Tard,	Trenton, N. J.
14	Mar. 11, 1826.	W. Day,	Gardiner, Me.
15	April 1, 1830.	T. M. Scott,	Falls Township, Pa.
16	May 4, 1831.	T. Hewitt,	Philadelphia, Pa.
17	June 13, 1831.	"	"
18	July 20, 1831.	Dyer & Richmond,	New Bedford, Mass.
19	May 19, 1832.	J. Aborn,	Trenton, N. J.
20	Dec. 16, 1833.	P. Farrell,	New York, N. Y.
21	Mar. 8, 1836.	J. Dunlap,	New Holland, Pa.
22	136 Mar. 3, 1837.	Moore and Bower,	Strasburgh, Pa.
23	265 July 11, 1837.	J. H. Tuck,	Nantucket, Mass.
24	1,824 Oct. 31, 1840.	J. Kirkman,	
25	2,405 Dec. 30, 1841.	Gamble and Hill,	Cincinnati, O.
26	2,600 April 30, 1842.	E. Marsh,	Alton, Ill.
27	4,389 Feb. 20, 1846.	J. Drummond,	New York, N. Y.
28	5,139 June 5, 1847.	B. F. Shelabarger,	Mifflintown, Pa.
29	5,576 May 16, 1848.	J. A. and A. F. Jones,	Lexington, Ky.
30	6,759 Oct. 2, 1849.	A. L. Brown,	New Haven, Conn.
31	7,033 Jan. 22, 1850.	H. Camp,	Dunkirk, N. Y.
32	7,559 Aug. 13, 1850.	J. G. Davis,	Buffalo, N. Y.
33	8,607 Dec. 23, 1851.	W. Humiston,	Troy, N. Y.
34	9,696 May 3, 1853.	G. Kendall,	Providence, R. I.
35	10,331 Dec. 20, 1853.	D. E. and M. Battershall,	Troy, N. Y.
36	10,730 April 4, 1854.	W. Humiston,	"
37	12,193 Jan. 9, 1855.	L. C. Ashley,	"
38	12,492 Mar. 6, 1855.	J. Stainthorp,	Buffalo, N. Y.
39	13,334 July 24, 1855.	W. Humiston,	Troy, N. Y.
40	13,973 Dec. 25, 1855.	L. C. Ashley,	"

No.	Date.	Inventor.	Residence.	
41	14,376	Mar. 4, 1856.	V. Squarza,	New York, N. Y.
42	14,662	April 15, 1856.	J. Jones,	Brooklyn, N. Y.
43	15,668	Sept. 2, 1856.	J. Robingson,	New Brighton, Pa.
44	15,821	Sept. 30, 1856.	B. D. Saunders,	Hollidays Cove, Va.
45	15,968	Oct. 28, 1856.	W. C. Childs,	Boston, Mass.
46	16,056	Nov. 11, 1856.	A. Hengstenberg,	Muscatine, Ia.
47	16,211	Dec. 9, 1856.	C. A. McPhetridge,	St. Louis, Mo.
48	16,754	Mar. 3, 1857.	B. D. Saunders,	Hollidays Cove, Va.
49	18,381	Oct. 13, 1857.	M. W. Brown,	Buffalo, N. Y.
50	21,706	Oct. 5, 1858.	J. H. Tatum,	New York, N. Y.
51	21,882	Oct. 26, 1858.	J. Jones,	Baltimore, Md.
52	22,592	Jan. 11, 1859.	J. H. Tatum,	New York, N. Y.
53	22,739	Jan. 25, 1859.	A. Meucci,	Clifton, N. Y.
54	22,921	Feb. 8, 1859.	H. Leonard,	New Bedford, Mass.
55	24,960	Aug. 2, 1859.	G. A. Stanley,	Cleveland, O.
56	25,227	Aug. 23, 1859.	S. R. Weeden,	
57	26,193	Nov. 22, 1859.	E. C. Leonard,	New Bedford, Mass.
58	26,429	Dec. 13, 1859.	H. Halvorson,	Cambridge, Mass.
59	26,440	Dec. 13, 1859.	Leonard and Ryder,	"
60	26,763	Jan. 10, 1860.	H. Halvorson,	"
61	26,780	Jan. 10, 1860.	Campbell Morfit,	New York, N. Y.
62	26,797	Jan. 10, 1860.	G. A. Stanley,	Cleveland, O.
63	26,798	Jan. 10, 1860.	"	"
64	27,763	April 3, 1860.	W. Thomas,	New York, N. Y.
65	27,995	April 24, 1860.	M. Massey,	Cleveland, O.
66	28,209	May 8, 1860.	G. A. Stanley,	"
67	28,210	May 8, 1860.	"	"
68	30,180	Sept. 25, 1860.	A. Meucci,	Clifton, N. Y.
69	29,416	July 31, 1860.	G. A. Stanley,	Cleveland, O.
70	29,506	Aug. 7, 1860.	F. Meyrose,	St. Louis, Mo.
71	29,729	Aug. 21, 1860.	J. H. Tatum,	New York, N. Y.
72	30,605	Nov. 6, 1860.	T. King,	Troy, N. Y.
73	{ Reissue } 1,131	Jan. 22, 1861.	W. Humiston,	"
74	{ Reissue } 1,132	Jan. 22, 1861.	"	"
75	35,359	May 27, 1862.	A. Black,	New York, N. Y.
76	36,798	Oct. 28, 1862.	G. Roth,	Cincinnati, O.
77	45,160	Nov. 22, 1864.	J. L. Klein,	New York, N. Y.
78	45,161	Nov. 22, 1864.	"	"
79	46,197	Jan. 31, 1865.	J. L. Field,	Lambeth, Eng.
80	50,000	Sept. 19, 1865.	C. Havard,	New York, N. Y.
81	{ Reissue } 2,108	Nov. 14, 1865.	W. Humiston,	Troy, N. Y.
82	{ Reissue } 2,190	Mar. 6, 1866.	"	"
83	59,460	Nov. 6, 1866.	H. Ryder,	New Bedford, Mass.
84	59,749	Nov. 20, 1866.	"	"
85	60,718	Jan. 1, 1867.	H. Grambo,	Philadelphia, Pa.
86	72,019	Dec. 10, 1867.	P. R. Gottstein,	Houghton, Mich.
87	86,059	Jan. 19, 1869.	E. Cowles,	Hounslow, Eng.
88	77,725	May 12, 1868.	J. L. Field,	Kensington, Eng.
89	88,779	April 13, 1869.	Field and Nation,	Lambeth, Eng.
90	97,749	Dec. 7, 1869.	M. Burlingame,	Garrattsville, N. Y.
91	101,857	April 12, 1870.	P. R. Gottstein,	Houghton, Mich.
92	103,388	May 24, 1870.	F. A. Taber,	Boston, Mass.
93	112,206	Feb. 28, 1871.	T. J. Barron,	Brooklyn, N. Y.
94	103,534	May 24, 1870.	Williams and Taber,	Boston, Mass.

	No.	Date.	Inventor.	Residence.
95	113,117	Mar. 28, 1871.	J. Wales,	New York, N. Y.
96	114,438	May 2, 1871.	C. Harvard,	"
97	120,105	Oct. 17, 1871.	H. Ryder,	New Bedford, Mass.
98	114,438	May 2, 1871.	C. Havard,	New York, N. Y.
99	119,785	Oct. 10, 1871.	J. A. Pease,	Catskill, N. Y.
100	125,632	April 9, 1872.	J. K. Truax,	Pittsburgh, Pa.
101	138,380	April 29, 1873.	I. Cole,	Brooklyn, N. Y.
102	138,925	May 13, 1873.	J. A. Pease,	Boston, Mass.
103	150,283	April 28, 1874.	C. N. Cadwallader,	Philadelphia, Pa.
104	172,423	Jan. 18, 1876.	P. R. Gottstein,	Houghton, Mich.
105	175,696	April 4, 1876.	C. F. Hall,	St. Louis, Mo.
106	{ Reissue } 7,777	July 3, 1877.	P. R. Gottstein,	Houghton, Mich.
107	214,258	April 15, 1879.	F. Maguire,	Cambridge, Mass.
108	241,810	May 24, 1881.	R. F. W. Loper,	Philadelphia, Pa.
109	242,630	June 7, 1881.	P. R. Gottstein,	Houghton, Mich.
110	243,272	June 21, 1881.	F. M. Joly,	Paris, France.
111	252,590	Jan. 17, 1882.	R. F. W. Loper,	Philadelphia, Pa.
112	258,778	May 30, 1882.	J. Livesey and D. Kidd,	London, Eng.
113	268,888	Dec. 12, 1882.	W. H. Haney,	Cincinnati, O.
114	269,664	Dec. 26, 1882.	"	"
115	269,885	Jan. 2, 1883.	A. A. Royeau,	Le Mans, France.
116	272,876	Feb. 27, 1883.	W. H. Haney,	Cincinnati, O.
117	276,602	May 1, 1883.	G. H. Kirk,	Philadelphia, Pa.
118	280,566	July 3, 1883.	J. Brelivet,	Enosburg Falls, Vt.
119	282,113	July 31, 1883.	G. P. Vicken,	Cincinnati, O.
120	284,486	Sept. 4, 1883.	J. B. Rodman and J. D. Bingham,	Leavenworth, Kan.
121	284,785	Sept. 11, 1883.	C. L. Werk,	Cincinnati, O.
122	298,488	May 13, 1884.	S. Clarke,	Childs Hill Works, Middlesex, Eng.
123	301,328	July 1, 1884.	F. Baumer,	Syracuse, N. Y.
124	305,787	Sept. 30, 1884.	J. Brelivet,	Enosburg Falls, Vt.
125	309,885	Dec. 30, 1884.	F. F. Schmitt,	Chicago, Ill.
126	310,874	Jan. 20, 1885.	H. L. Brevoort,	Brooklyn, N. Y.
127	314,347	Mar. 24, 1885.	G. Roth,	Cincinnati, O.
128	319,156	June 2, 1885.	C. L. Werk,	Westwood, O.
129	323,058	July 28, 1885.	J. B. Mitchell,	Portland, Me.
130	329,536	Nov. 3, 1885.	S. Clarke,	Childs Hill Works, Middlesex, Eng.
131	330,200	Nov. 10, 1885.	A. F. Baumer,	Syracuse, N. Y.
132	333,013	Dec. 22, 1885.	L. Homan,	Cincinnati, O.
133	343,567	June 15, 1886.	S. Clarke,	Childs Hills Works, Middlesex, Eng.
134	345,272	July 13, 1886.	E. L. Brown,	Chicago, Ill.
135	370,562	Sept. 27, 1887.	F. F. Schmitt,	"



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